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A PORTABLE APPARATUS FOR DETERMINING THE RELATIVE WEAR RESISTANCE OF CONCRETE FLOORS

By Louis Schuman and John Tucker, Jr.

ABSTRACT

A portable machine for producing rapid wear and an optical gage for measuring the depths of wear were designed and built. Studies were made on 138 slabs of concrete to determine the effect of various mixes and *C/W* ratios and of such factors as aggregate types and grading, finishing procedures, dust coats, and liquid surface treatments. The tests show that metallic hardeners, dust coats containing cement, and delayed troweling are effective in increasing wear resistance. The test methods used show advantages in the use of dry mixes, coarse aggregates, and of adequate damp curing. The use of a high-early-strength cement permitted the damp curing period to be greatly reduced without decreasing the wear resistance.

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I. INTRODUCTION

Wear tests of concretes have been made by many investigators [1 to 7].[1] Abrams [2], using the Talbot-Jones rattler, determined the effect of such factors as the water content and the fineness modulus of aggregates on the wear to depths of 2 in. In tests made by the Bureau of Public Roads [4], rubber-tired wheels with chains were used for abrading the surfaces under test; these studies were concerned mainly with the wear resistance of various aggregates. The effect of methods

[1] Figures in brackets indicate the literature references at the end of this paper.

of finishing and of other factors upon the wear resistance of the surface "skin," that is, to a depth of approximately 0.1 in., were not studied.

The purpose of the present investigation was to develop an apparatus for producing rapid wear and a method for measuring depths of wear up to 0.1 in.

II. APPARATUS

1. MACHINE FOR PRODUCING RAPID WEAR

The machine which was developed is shown in figure 1. It consists essentially of three vertical shafts to which the 3-in.-diameter cold-rolled steel abrading disks *A*, *B*, and *C* are attached by means of universal joints. Plate *P*, carrying these abrading elements, rotates at 2 rpm, while the abrading disks rotate at 180 rpm. Cups *L* attached to the disk shafts were loaded with lead to give a total load on each disk of 25 lb. The net abrading area (face, less slots) of each disk was 5.6 in.^{2}; the pressure was therefore 4.4 lb/in.^{2}.

The revolving disks produced measurable wear; but for more rapid wear an abrasive, No. 60 silicon carbide grain, was used. This was fed from cup *D*, through a tube with $\frac{1}{16}$-in. diameter opening, and deposited on the surface to be abraded, about $\frac{1}{4}$ in. within the outer edge of the track formed by the abrading disks. The rotation of plate *P* and of the disks causes the abrasive to be swept gradually toward the center and, together with particles loosened from the test slab, to pile up near the plate support. The circular track formed by the simultaneous rotation of the plate and disks has an inside diameter of 12 in. and an outside diameter of 18 in. Preliminary tests showed that, with the loads and abrasive used, depths of wear up to about 0.1 in. could be obtained when the machine was operated for 20 minutes. By measuring the depth of wear after several shorter periods of operation of the machine, it was possible to study the effects of methods of finishing, dust coats, and "hardeners."

2. DEVICE FOR MEASURING DEPTH OF WEAR

The device used for measuring the average depth of the abraded surface is shown diagrammatically in figure 2. The light from a straight-filament 35-watt galvanometer lamp, *A*, is reflected by a flat chromium-plated reflector, *B*. Though reflection takes place over the whole area of the reflector, the light beam which just passes the straight edge, *E*, placed across the abraded surface affords a measure of the depth of wear. The beams above this "measuring beam" will not appear beyond the straight edge; the beams below it illuminate the worn surface up to a contour, *C*, which is a magnified profile of a radial section of the worn surface. The distance, *m*, from straight edge *E* to any point, *P*, on this contour *C* is read on a scale, *S*, ruled on a celluloid plate and graduated in 0.1 in. The magnification is the distance, *d*, divided by the height, *h*, above the original surface. Thus the depth of wear at a point, *P*, equals $m \div d/h$. The average distance of contour *C* from straight edge *E* was estimated by inspection and used to calculate the depth of wear. The distances *h* and *d* were measured directly; in the device used the magnification d/h was 32, and one division on the scale corresponded to a depth of wear of 0.1/32, or 0.003 in.

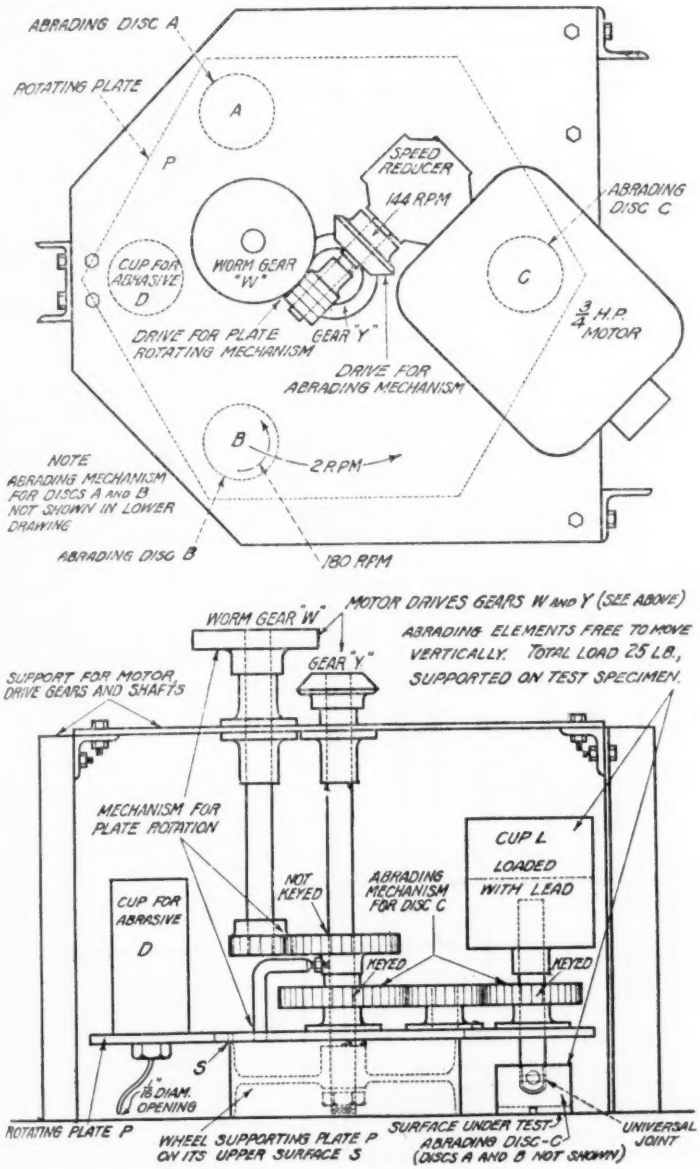

FIGURE 1.—*Abrasion apparatus.*

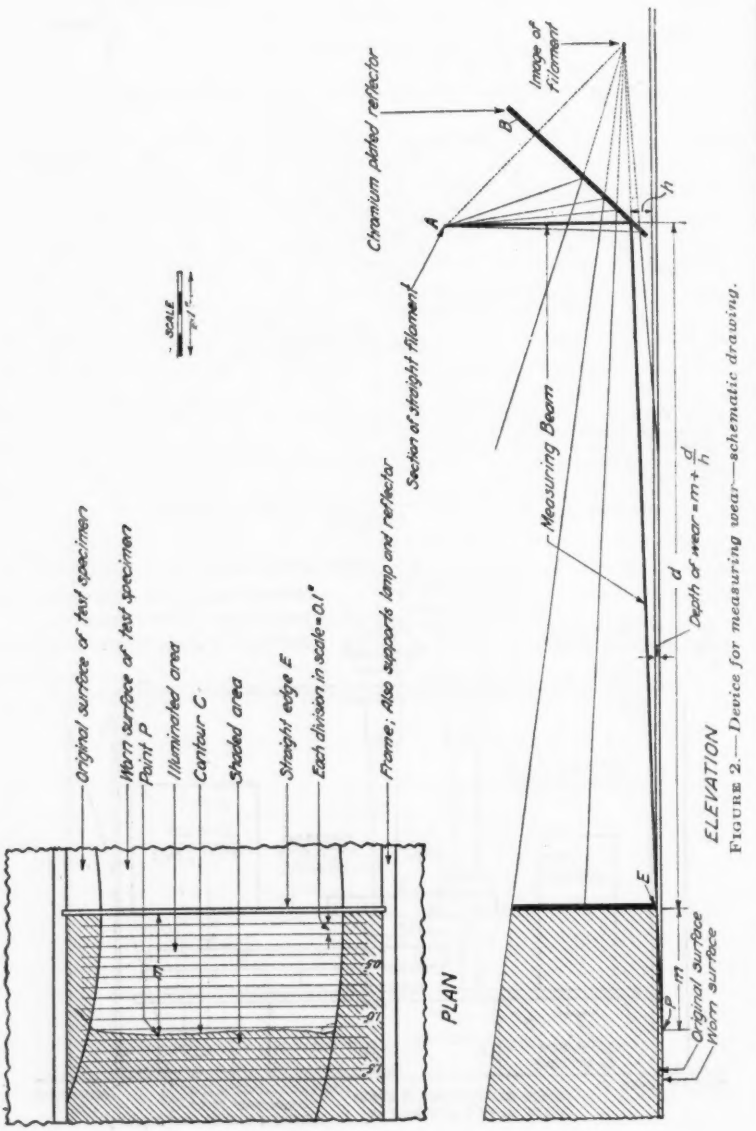

FIGURE 2.—*Device for measuring wear—schematic drawing.*

III. OUTLINE OF INVESTIGATION

The reliability of the apparatus for determining the relative resistance to wear of various surfaces was studied by means of tests on a series of concrete slabs of mixes varying from a 1:2 mortar to a 1:3:6 concrete, in which various cements, *C/W* ratios, sands, and coarse aggregates were used. The surfaces were finished by different methods, such as the application of dust coats of cement, cement and sand, or cement and metallic aggregate. The effects of various time intervals between placing and troweling, and of variation in periods of damp curing were also studied. The effects of sodium silicate and magnesium fluosilicate solutions, two types of materials frequently used for the treatment of concrete surfaces, were studied.

IV. MATERIALS, TEST SPECIMENS, AND TEST METHODS

1. MATERIALS

A "normal" portland cement meeting the requirements of Federal Specification SS–C–191a was used in most of the specimens. This cement contained 2.5 percent of Fe_2O_3 and 6.9 percent of Al_2O_3. Three slabs were made with a cement also meeting the requirements of the specification but with an Fe_2O_3 content of 4.6 percent and an Al_2O_3 content of 4.2 percent, and eight slabs were made with a high-early-strength cement meeting the requirements of Federal Specification SS–C–201.

The gradings of the various aggregates used are shown in table 1. Sands *A*, *B*, *C*, *D*, and *E* and the gravels, all from the Potomac River, were largely silicious. The "traprock" sand (*F*) and the No. 8 to ¼-in. "traprock" were from a Pennsylvania quarry. The No. 8 to ⅜-in. crushed limestone was from Martinsburg, W. Va.

TABLE 1.—*Grading of aggregates*

[Percentage retained on various sieves]

Sieve	Fine aggregate							Coarse aggregate				
	Potomac River sand					"Traprock" sand, *F*	Metallic aggregate[b]	Potomac River gravel			Crushed limestone, No. 8 to ⅜ in.	Crushed "traprock", No. 8 to ½ in.
	A	*B*	*C*	*D*[a]	*E*[a]			No. 8 to ¼ in.	¼ in. to ⅜ in.	¼ in. to ¾ in.		
	%	%	%	%	%	%	%	%	%	%	%	%
¼ in.	0	0	0	0	0	0	0	0	0	0	0	0
⅜ in.	0	0	0	0	0	0	0	0	0	50	0	16
No. 4	1	1	4	6	0	1	0	61	100	100	66	72
No. 8	9	7	27	56	0	16	0	94	100	100	99	98
No. 16	16	20	43	100	0	35	6	99	100	100	99	100
No. 30	37	28	57	100	0	50	49	100	100	100	99	100
No. 50	86	80	79	100	78	66	94	100	100	100	100	100
No. 100	98	98	98	100	97	81	99	100	100	100	100	100
Fineness modulus	2.47	2.44	3.08	4.62	1.75	2.49	2.48	5.54	6.00	6.50	5.63	5.86

[a] Prepared from sand *A* by screening.
[b] Metallic aggregate is to be distinguished from so-called metallic waterproofing compounds, the former being coarser and free of salts, as well as of oil, grease, and nonferrous metals.

In the study of liquid surface treatments, three grades of sodium silicate were used: The first is designated water glass and was not analyzed. The second had a soda-silica ratio of 1:3.13 and is designated high-silica water glass. The third had a soda-silica ratio of 1:1.6 and is designated low-silica water glass. Each of the three grades was diluted to an 11-percent solution by weight. The magnesium fluosilicate "hardener" solution was obtained in two strengths—a 15-percent solution (based on the hydrated salt $MgSiF_6.6H_2O$) which was applied without diluting, and a 22-percent solution which was diluted to various concentrations before application.

2. PREPARATION AND TREATMENT OF TEST SPECIMENS

The test specimens were slabs 23.5 by 27.5 in., usually 1 in. thick; when the aggregate coarser than ½ in. was used, slabs 2 in. thick were made. The materials were stored and the specimens made in a laboratory maintained at 70°±2° F. The materials were mixed in either a laboratory batch mixer or a small concrete drum mixer. Mixing was in all cases continued for 3 minutes after all the ingredients had been placed in the drum, because this time was required for the drier mixes. Flow values on the 10-in. flow table were determined on mixes with aggregates up to ½ in. On those with the coarser aggregate, slumps were determined.

The steel mold for casting the slab was placed over a sheet of asphalt impregnated roofing felt spread on the laboratory floor. The prepared mortar or concrete was placed in the mold, and spread and compacted with a mixing trowel. A wooden straightedge was then used to screed and further compact the material, the edges of the mold acting as a guide. Where a smooth surface could not be obtained by screeding, the surface was further smoothed with a wooden float. Final finishing with a steel trowel was done after various time intervals. Troweling was continued only long enough in each case to produce a smooth surface.

The specimens were kept in the molds in the laboratory for 24 hours. After this period the specimens not damp-cured were air-stored in the same laboratory until tested. The damp-cured slabs were placed in the damp room maintained at 70°±2° F and 95-percent or greater relative humidity. After damp-curing, these were also air-stored in the laboratory. All slabs except those made with high-early-strength cements were at least 28 days old when tested

The materials, mix proportions, methods of finishing, and the curing treatments are given in table 2.

TABLE 2.—*Test results*

Slab No.[a]	Proportions by weight, dry materials	Aggregates	C/W by weight	Total water content: Gallons per bag	Total water content: Volume[b]	Flow, 10-in. table, 25 half-in. drops	Interval between placing and troweling	Duration of storage: Damp	Duration of storage: Air	Surface treatment	Wear test results: Test *D1* and *D2*, 5 minutes, no abrasive[c]: Weight of dust[d]	Wear test results: Test *D1* and *D2*, 5 minutes, no abrasive[c]: Depth of wear[e]	Wear test results: Tests with abrasive. Depth of wear during each test: Test *A1*, 10 min[f]	Wear test results: Tests with abrasive. Depth of wear during each test: Test *A2*, 2d 10 min[g]	Degree of pitting[h]	Remarks
	Ratio		*Ratio*	*Gal*	%	%	*Hours*	*Days*	*Days*		*g*	*0.001 in.*	*0.001 in.*	*0.001 in.*		
1	1:2	Sand *A*	2.05	5.5	30.8	110	4	6	21		31	7	17	24	*M*	Water on surface after troweling.
2	1:2	Sand *E* (<No. 30)	2.05	5.5	30.8	75	2.5	6	21		34	6	22	33	*E*	Readily finished.
3	1:2	Sand *A*	2.26	5.0	28.6	75	2	6	21		40	8	16	15	*M*	Do.
4	1:2	do	2.26	5.0	28.6	75	2	6	21		34	8	19	16	*M*	Do.
5	1:2	do	2.26	5.0	28.6	75	5	6	21		12	2	12	10	*S* to *M*	Do.
6	1:2	do	2.26	5.0	28.6	75	2&5	6	21		22	7	14	16	*M*	Do.
7	1:2	do	2.26	5.0	28.6	75	0	6	21	Dry cement, 3/8 lb/ft²	19	6	8	6	*S*	Do.
8	1:2	do	2.26	5.0	28.6	75	2.5	6	21	3 coats water glass	34	7	20	14	*E*	Compare with slabs 3 and 4.
9	1:2	do	2.26	5.0	28.6	75	2	6	21	3 coats magnesium fluosilicate 15%.	37	9	24	19	*M*	Do.
10	1:2	do	2.26	5.0	28.6	75	2	6	21	2 coats cement-wash; wet 1 week, dry 2 weeks.	44	6	21	17	*M*	Do.
11	1:2	do	2.50	4.5	26.6	40	2.5	6	21		12	4	9	13	*M*	Fairly dry; readily finished.
12	1:2	do	2.50	4.5	26.6	40	2	6	21		18	3	13	15	*M*	Do.
13	1:2	do	2.50	4.5	26.6	40	2	0	28		25(12)	6	19	25	*E*	Do.
14	1:2	do	2.50	4.5	26.6	40	2	3	24		11	3	9	13	*M*	Do.
15	1:2	do	2.50	4.5	26.6	40	3	6	21		11	2	9	11	*M*	Do.

[a] Slabs marked (2) made in duplicate, marked (3) in triplicate.

[b] Water content: $\frac{\text{Volume of mixing water}}{\text{Sum of absolute volumes of components}} \times 100$.
Specific gravity of cement, 3.1.
Specific gravity of aggregate, 2.6.

[c] Values for *D2* test in brackets; all others *D1*.

[d] Standard deviation individual slabs, 4.4 g.
[e] Standard deviation individual slabs, 0.0012 in.
[f] Standard deviation individual slabs, 0.0025 in.
[g] Standard deviation individual slabs, 0.0025 in.
} See text for method of computation.

[h] *N* = Negligible
S = Slight
M = Moderate
E = Excessive
} See figures 3, 4, and 5.

TABLE 2.—*Test results*—Continued

Slab No.	Proportions by weight, dry materials	Aggregates	C/W by weight	Total water content		Flow, 10-in. table, 25 half-in. drops	Interval between placing and troweling	Duration of storage		Surface treatment	Wear test results				Degree of pitting	Remarks
				Gallons per bag	Volume			Damp	Air		Test *D1* and *D2*, 5 minutes, no abrasive		Tests with abrasive. Depth of wear during each test			
											Weight of dust	Depth of wear	Test *A1*, 10 min	Test *A2*, 2d 10 min		
	Ratio		*Ratio*	*Gal*	*%*	*%*	*Hours*	*Days*	*Days*		*g*	*0.001 in.*	*0.001 in.*	*0.001 in.*		
16	1:2	Sand *D* (>No. 16)	2.50	4.5	26.6	80	2	6	21		23	3	7	7	*S*	Fairly wet mix.
17	1:2	Sand *A*	2.50	4.5	26.6	40	0	6	21	Dry cement, ¼ lb/ft [1]	20	7	4	9	*S*	Fairly dry; readily finished.
18	1:2	do	2.50	4.5	26.6	40	2	6	21	do	11	3	4	7	*S*	Do.
19	1:2	do	2.50	4.5	26.6	40	1.5	6	21	1:1 cement; SIC No. 60, ½ lb/ft.[2]	56	13	11	13	*S* to *M*	Do.
20	1:2	Limestone, No. 8 to ¼ in.	2.50	4.5	26.6	40	4	6	21		24	7	12	14	*M*	Difficult to trowel.
21	1:2	Sand *A*	2.82	4.0	24.4	10	1	6	21		6	3	5	6	*S*	Very dry mix.
22	1:2	Gravel, No. 8 to ¼ in.	2.82	4.0	24.4	30	2	6	21		21	7	7	10	*S*	Somewhat sticky when troweled.
23	1:2	do	2.82	4.0	24.4	30	3	6	21		14	4	10	10	*S*	Do.
24	1:2.5	Sand *A*	1.88	6.0	29.1	95	3.5	6	21		62	12	27	26	*E*	Water on surface after troweling.
25	1:2.5	do	1.88	6.0	29.1	95	5	6	21		22	3	16	13	*M*	Readily finished.
26	1:2.5	do	2.05	5.5	27.4	55	2.5	6	21		48	8	21	21	*E*	Fairly dry; readily finished.
27	1:2.5	do	2.05	5.5	27.4	55	4	6	21		10	2	12	16	*M* to *E*	Difficult to trowel at 4 hr.
28	1:2.5	do	2.26	5.0	25.5	35	2	6	21		8	6	6	9	*S*	Very dry mix.
29	1:2.5	Gravel, No. 8 to ¼ in.	2.82	4.0	21.6	45	1.5	6	21		21	6	14	8	*S*	Difficulty in smoothing surface.
30	1:3	Sand *A*	1.61	7.0	29.4	85	4	6	21		74(30)	14	33	25	*E*	Water on surface after troweling.
31	1:3	do	1.61	7.0	29.4	85	5	6	21		24	6	17	19	*M* to *E*	Some water on surface after troweling.
32	1:3	do	1.61	7.0	29.4	85	2	6	21	Dry cement, 1¼ lb/ft [1]	11	2	8	9	*N*	Badly crazed surface.
33	1:3	do	1.61	7.0	29.4	85	3	6	21	1:1 cement:sand, ½ lb/ft.[2]	17	7	15	16	*S*	Readily finished.
34	1:3	do	1.73	6.5	27.9	60	2.5	6	21		40	9	29	25	*E*	Fairly dry; readily finished.
35	1:3	do	1.73	6.5	27.9	60	2	6	21	Dry cement, ¼ lb/ft [1]	7	3	12	16	*M*	Do.

34	1:3	....do	1.73	6.5	27.9	60	2.5	6	21		40	9	29	25	*E*	Fairly dry; readily finished.
35	1:3	....do	1.73	6.5	27.9	60	2	6	21	Dry cement, ¼ lb/ft.²	7	3	12	16	*M*	Do.
36	1:3	....do	1.73	6.5	27.9	60	2	6	21	2:1 metal:cement, 0.45 lb/ft.²	13	4	4	3	*M*	Do.
37	1:3	....do	1.73	6.5	27.9	60	2	6	21	2:1 metal:cement, 0.90 lb/ft.²	19	3	1	2	*N*	Lowest wear all slabs.
38	1:3	....do	1.73	6.5	27.9	60	2	6	21	2:1 metal:cement, 3% oil, 0.45 lb/ft.²	16	3	6	12	*E*	Oil causes pitting.
39	1:3	....do	1.73	6.5	27.9	60	5	6	21		17	3	14	16	*M* to *E*	Fairly dry; readily finished.
40	1:3	....do	1.88	6.0	26.3	30	2	6	21		14	3	16	18	*M*	Dry; somewhat difficult to finish.
41	1:3	....do	1.88	6.0	26.3	30	2 & 4	6	21		9	2	11	15	*M*	Dry mix.
42	1:3.5	....do	1.88	6.0	24.0	f 60	0	6	21		6	5	4	17	*E*	Very dry mix.
43	1:4	....do	1.61	7.0	24.8	f 50	0	6	21		10	2	17	35	*E*	Do.
44	1:2	Gravel, ⅜ in.	2.82	4.0	24.4	55	3	6	21		18	10	9	14	*S*	Difficult to trowel.
45	1:1:2	Sand *A* and ⅜-in. gravel.	2.26	5.9	23.0	65	4	6	21		16	8	11	8	*S*	Fairly dry when finished.
46	1:1:2	....do	2.50	4.5	21.2	40	2.5	6	21		17	7	15	8	*S*	Somewhat wet.
47	1:1:2	....do	2.50	4.5	21.2	40	4	6	21		15	3	11	8	*S*	Somewhat wet; difficult to finish.
48	1:1:2	....do	2.50	4.5	21.2	40	2.5	6	21	Scraped with leather belting.	33	11	15	10	*S*	Do.
49	1:1:2	....do	2.50	4.5	21.2	40	2	6	21	2:1 metal: cement, 0.45 lb/ft.²	11	5	3	3	*S*	Difficult to trowel.
50	1:1:2	....do	2.82	4.0	19.3	25	2.5	6	21		18	3	12	9	*S*	Dry mix.
51	1:1:2.5	....do	2.50	4.5	19.2	30	2.5	6	21		17	3	12	9	*S*	Difficult to trowel; undersanded.
52	1:1:3	....do	2.50	4.5	17.6	f 50	1.5	6	21		14	4	10	8	*S*	Do.
53	1:1.5:2	....do	2.05	5.5	22.5	80	4	6	21		18	5	13	11	*S*	Somewhat wet.
54	1:1.5:2	....do	2.05	5.5	22.5	80	5	6	21		17	5	14	10	*S*	Do.
55	1:1.5:2	....do	2.26	5.0	20.9	35	2	6	21		8	4	11	7	*S*	Readily placed and troweled.
56	1:1.5:2	....do	2.26	5.0	20.9	35	2 & 5	6	21		15	3	7	9	*S*	Do.
57	1:1.5:2	Sand *A* and ⅜-in. limestone.	2.26	5.0	20.9	30	3	6	21		24	6	12	10	*S*	Fairly dry; readily finished.
58	1:1.5:2	Sand *A* and ⅜-in. gravel.	2.50	4.5	19.2	f 35	2	6	21		13	2	9	9	*S*	Fairly dry; easily finished.
59	1:1.5:3	....do	2.05	5.5	19.0	40	2	6	21		22	8	17	12	*S*	Slightly wet; easily finished.
60	1:1.5:3	....do	2.05	5.5	19.0	40	3	6	21		28	7	14	10	*S*	Do.
61	1:1.5:3	....do	2.26	5.0	17.7	f 25	2	6	21		18	3	17	9	*S*	Dry mix; difficult to trowel.
62	1:2:2	....do	2.05	5.5	20.6	30	2	6	21		15	5	17	11	*M*	Slightly wet; readily finished.

f Flow on "dry" side: See text.

TABLE 2.—*Test results*—Continued

Slab No.	Proportions by weight, dry materials	Aggregates	C/W by weight	Total water content: Gallons per bag	Total water content: Volume	Flow, 10-in. table, 25 half-in. drops	Interval between placing and troweling	Duration of storage: Damp	Duration of storage: Air	Surface treatment	Wear test results: Test D1 and D2, 5 minutes, no abrasive: Weight of dust	Wear test results: Test D1 and D2, 5 minutes, no abrasive: Depth of wear	Wear test results: Tests with abrasive. Depth of wear during each test: Test A1, 10 min	Wear test results: Tests with abrasive. Depth of wear during each test: Test A2, 2d 10 min	Degree of pitting	Remarks
	Ratio		*Ratio*	*Gal*	*%*	*%*	*Hours*	*Days*	*Days*		*g*	*0.001 in.*	*0.001 in.*	*0.001 in.*		
63	1:2:3	Sand *A* and ¾-in. gravel	1.88	6.0	19.0	[f] 45	2	6	21		25	9	20	12	*M*	Fairly dry; somewhat difficult to finish.
64	1:2:3	do	1.88	6.0	19.0	[f] 45	3	6	21		33	7	13	10	*M*	Do.
65	1:2:3	do	2.05	5.5	17.7	40	2	6	21		17	6	13	12	*S*	Do.
66	1:2:4	Sand *A* and ¾-in. gravel.	1.73	6.5	17.8	[h] 0.3	4	6	21		48	9	16	12	*S*	Considerable floating required.
67	1:3:6	do	1.33	8.5	16.5	[h] 0.5	4	6	21		57	14	22	13	*S*	Do.
68	1:3:6	do	1.33	8.5	16.5	[h] 0.5	3	6	21	1:2 cement: sand, 0.5 lb/ft.[2]	19	4	16	15	*S*	Dust coat aids finishing.
69(3)	[i] 1:2	Sand *B*	2.50	4.5	26.6	60	2	6	21		22	3	18	13	*M*	Fairly dry; readily finished.
70(2)	[m] 1:2	do	2.50	4.5	26.6	40	2	0	14		16	3	15	13	*M*	Do.
71(3)	[m] 1:2	do	2.50	4.5	26.6	30	2	2	10		14	4	10	13	*M*	Do.
72(3)	[m] 1:2	do	2.50	4.5	26.6	35	2	6	10		15	4	11	11	*M*	Do.
73(2)	1:2	do	2.50	4.5	26.6	40	2	0	28		64	13	32	27	*E*	Do.
74(2)	1:2	do	2.50	4.5	26.6	40	2	2	25		27	5	20	19	*M*	Do.
75(2)	1:2	do	2.50	4.5	26.6	45	2	6	21		25	5	16	12	*M*	Compare with No. 96.
76(2)	1:2	do	2.50	4.5	26.6	45	2	27	14		13	4	12	11	*S*	Compare with No. 75.
77(2)	1:2	do	2.50	4.5	26.6	45	2	60	14		8	3	7	8	*S*	Do.
78(2)	1:2	do	2.26	5.0	28.6	100	2	6	21		28(4)	6	15	11	*M*	Compare with No. 97, 98, and 99.
79(2)	1:2	do	2.26	5.0	28.6	95	2	0	28		81(56)	15	45	26	*E*	Compare with No. 100, 101, 102, and 103.
80(2)	1:2	Sand *C*	2.50	4.5	26.6	85	2	6	21		27(3)	6	14	11	*M*	Somewhat wet.
81(2)	1:2	do	2.50	4.5	26.6	75	4.5	6	21		10(3)	2	10	10	*S*	Do.
82(2)	1:2	do	2.82	4.0	24.4	20	2	6	21		11	2	11	12	*M*	Very dry mix.
83(2)	1:2.5	do	2.50	4.5	23.6	25	2	6	21		14	4	14	14	*M*	Do.
84(2)	1:3	do	1.88	6.0	26.3	100	5	6	21		41(6)	12	21	16	*M*	Rather wet; compare with No. 40.

85(2)	1:3	do	2.05	5.5	23.7	30	2	6	21		17	5	18	16	*M*	Very dry mix.
86(2)	1:1:2	Sand *C* and ⅜-in. gravel.	2.50	4.5	21.2	40	2	6	21		16	6	15	10	*S*	Dry; difficult to trowel; undersanded.
87(2)	1:1:2	do	2.26	5.0	23.0	85	2 & 5	6	21		17	4	19	11	*S*	Wet mix.
88(2)	1:1.5:2	do	2.26	5.0	20.9	40	2 & 5	6	21		15	4	14	9	*S*	Fairly dry; difficult to finish.
89(2)	1:1.5:2	do	2.05	5.5	22.5	80	2 & 5	6	21		13	4	19	12	*S*	Fairly wet.
90(2)	1:1.5:2	do	2.05	5.5	22.5	85	5	6	21		17(1)	5	20	11	*S*	Do.
91(2)	1:2:2	do	2.05	5.5	20.6	60	2	6	21		45	12	18	14	*S*	Difficult to float and trowel.
92	1:2:3	Sand *C* and ¾-in. gravel.	1.88	6.0	19.0	[h] 1.1	2 & 5	6	21		25(1)	4	20	12	*S*	Fairly wet; easily placed and troweled.
93	1:2:4	do	1.73	6.5	17.8	[h] 0.5	2	6	21		69(2)	18	24	12	*S*	Difficult to trowel; wet surface.
94	1:1.5:2	Sand *F* and ½-in. traprock.	2.05	5.5	22.5	30	4	6	21		30	8	17	10	*S*	Do.
95(2)	1:2:2	do	1.88	6.0	22.1	40	4	6	21		55	15	19	11	*S*	Difficult to finish; wet surface.
96(2)	1:2	Sand *B*	2.50	4.5	26.6	40	2	6	21	3 coats high silica water glass.	19	4	13	13	*M*	Compare with No. 75.
97(2)	1:2	do	2.26	5.0	28.6	90	2	6	21	do	27(5)	6	20	18	*M*	Compare with No. 78.
98(2)	1:2	do	2.26	5.0	28.6	95	2	6	21	3 coats low silica water glass.	27(4)	8	16	16	*M*	Do.
99(2)	1:2	do	2.26	5.0	28.6	85	2	6	21	3 coats magnesium fluosilicate.	27(5)	6	15	13	*M*	Do.
100(2)	1:2	do	2.26	5.0	28.6	100	2	0	28	3 coats water	79(17)	15	30	22	*M*	Compare with No. 79.
101(2)	1:2	do	2.26	5.0	28.6	95	2	0	28	3 coats high silica water glass.	66	14	32	31	*E*	Compare with No. 79 and 100.
102(2)	1:2	do	2.26	5.0	28.6	95	2	0	28	3 coats low silica water glass.	51(22)	10	26	24	*E*	Do.
103(2)	1:2	do	2.26	5.0	28.6	80	2	0	28	3 coats magnesium fluosilicate.	53(31)	10	30	30	*E*	Do.
										Maximum	81(56)	18	45	33		
										Minimum	6(1)	2	1	2		

[f] Flow on "dry" side: See text.
[h] Slump, inches.
[i] "High iron" cement.
[m] High-early-strength cement.

The liquid "hardeners" were applied to 15 slabs of a 1:2 mortar when the slabs were 28 days old. The water-glass solutions were applied in three coats, on successive days, each solution being applied liberally with a brush. The slabs were not scrubbed with water between coats, as usually recommended [1], since such scrubbing might have the effect of additional damp-curing. After the last coat, the specimens were air-stored 2 weeks before testing.

The magnesium fluosilicate solutions were also applied in three coats. The 15-percent solution was applied without dilution. The 22-percent solution was diluted with two volumes of water for the first coat, with one volume for the second coat, and with one-half volume for the third coat. The second and third coats of the fluosilicate solutions were absorbed better than the corresponding coats of water-glass solutions, as might be expected.

To determine any possible effect of the water in the solutions on specimens not previously damp-cured, two slabs of mix 100 were given three surface applications of water, in amounts about equal to the amounts of magnesium fluosilicate or of water-glass solutions applied to the other slabs.

Most of the specimens with sand *C* and those made for comparing cements, curing periods, and liquid "hardener" treatments were made in duplicate or in triplicate, as indicated in table 2. It was not considered necessary to make duplicate or triplicate specimens in other cases.

3. TEST METHODS

Preliminary tests had shown that a 5-minute test without abrasive might give information of value, since the surface layer or "skin" was often more easily abraded than the subsurface. Consequently, all of the test slabs were first abraded by this method, which is designated test *D1*. The loosened dust was brushed off and weighed, and the depth of wear measured at eight equally spaced positions.

Two 10-minute tests, designated *A1* and *A2*, were then made on the same section of the slab, using abrasive; and the depth of wear for each test was measured.

Twenty-eight slabs were given a further 5-minute test without abrasive, which is designated *D2*, on the area already abraded in tests *D1*, *A1*, and *A2;* and the weight of dust was determined as in test *D1*.

The degrees of pitting (table 2) were estimated by inspection.

The amount of dust removed in each of six successive half-minute tests from a section of the surface not abraded in the previous tests was determined for the slabs treated with liquid hardeners and for the corresponding untreated specimens (slabs 78, 79, and 97 to 103).

The reproducibility of results in table 2 is illustrated by like slabs 3 and 4 and also slabs 11 and 12. Individual test data are not given on the remainder of the slabs made in duplicate or triplicate. To furnish information on the reproducibility of the results obtained upon like slabs, the dispersion for the individual slab was measured, as expressed by the standard deviation, and computed from the formula:

$$\sigma = \sqrt{\frac{\Sigma d^2}{N-n}},$$

where d is the deviation of the average value for a slab from the average for a like group (2 or 3 slabs in this case), n is the number of groups of like slabs, and N is the total number of slabs in these groups.

The reproducibility of the results is as follows:

Test	Standard deviation
D1—weight of dust in grams	4.4
D1—depth of wear in 0.001 in	1.2
A1—depth of wear in 0.001 in	2.5
A2—depth of wear in 0.001 in	2.5

Computations were made also of the deviations in the eight individual readings on a single slab. These deviations are given in the following tabulation under the head of standard deviation of the individual measurement. These computations were made for a group of 10 representative slabs:

Test	Standard deviation	
	Individual measurement	Slab
D1—depth of wear in 0.001 in	2.5	1.0
A1—depth of wear in 0.001 in	5.5	2.0
A2—depth of wear in 0.001 in	6.5	2.3

In the last column of the tabulation is given the theoretical standard deviation of a single slab computed from the standard deviation of the individual measurements. This standard deviation of the single slab is the standard deviation of the individual measurement divided by the square root of the number of individual measurements made on one slab—eight in this case.

From the values of the theoretical standard deviation of the slab and the standard deviation computed for like slabs in the first tabulation, it is at once apparent that variations between different slabs are of the same magnitude as would be caused by the variations in the individual readings on a single slab and that results on duplicate slabs do not differ significantly from each other.

V. TEST RESULTS

1. GENERAL DISCUSSION

The weights of dust removed in test *D1* vary from 6 to 81 g, and the depths of wear in the same test from 0.002 to 0.018 in. In test *A1* the depths of wear varied from 0.001 to 0.045 in., and in test *A2* from 0.002 to 0.035 in. The values for test *D2* are generally considerably lower than for test *D1*.

The relative depths of wear in tests *D1* and *A1* depend on the mix, finishing procedure, and other factors, especially surface treatment. For most of the tests, the depth of wear for test *A1* is from two to five times as much as for test *D1*. In 10 cases, however, the depth for test *A1* differs little from or is less than that for test *D1*, indicating a highly resistant subsurface compared with the surface layer (for

example, Nos. 7, 17, 28, 37). Where the values for the *D1* tests are high, exceeding 0.011 in., indicating a comparatively poor surface layer, the subsurface may be comparatively resistant, with values in the *A1* test less than 0.023 in. (slabs 48, 67, 91, 95).

For most of the slabs, the wear in test *A2* was approximately equal to or less than that in test *A1*; however, for a few of the slabs it was considerably higher (slabs 2, 42, 43).

2. EFFECT OF VARIOUS FACTORS ON WEAR

(a) WATER CONTENT AND GRADING OF AGGREGATE

Most of the mixes were moderately dry, although some were sufficiently wet to have flow values as high as 110. As is known, decrease in water content will decrease the flow-table reading to a certain point, which we will term the minimum flow, beyond which further decrease in water content will cause increase in flow readings. Thus, two mixes with very different water contents may give equal flow readings, one of the mixes being fairly wet, the other dry. The dry mixes are indicated in table 2.

Most of the mixes used could be finished readily by hand floating and troweling. In some cases, however, difficulty was encountered especially where there appeared to be an excess of coarse aggregate or insufficient sand—for example, specimens 51 and 52. The amount of floating and troweling necessary in such cases sometimes brought free water to the surface (slabs 93, 94, and 95). The wetter mortar mixes also showed surface water, especially if troweled too soon (slabs 1, 24, 30). Although excess water was not the only cause of poor wear resistance, its appearance on the slabs, as noted in table 2, usually was followed by poor wear resistance in the *D1* test, for example, slabs 24, 30, 93, 94, and 95.

Figure 3 shows the total depth of wear, after completion of tests *D1*, *A1*, and *A2*, plotted for mortars and concretes of various proportions and the C/W ratios in which sand *A* or *C*, with or without ⅜- or ¾-in. gravel, was used. For any particular mix proportions the depth of wear decreases in general with increasing C/W ratios. For example, the 1:2 mortar shows a decrease from 0.048 to 0.014 in., as the C/W ratio is increased from 2.05 to 2.82. For the slabs containing gravel there is also a decrease, though relatively smaller.

For a particular C/W ratio the total depth of wear of the mortar mixes, in three out of five cases, decreases considerably as the proportion of the sand increases, in the other two cases showing little change (fig. 3). For the mixes containing gravel the depth of wear is lowest for the 1:1.5:2 mix. For both sand *C* and sand *A* the depth of wear increases as the gravel mixes become leaner in cement. The 1:2 mix, $C/W=2.26$, showed about twice the depth of wear obtained on the 1:1.5:2 mix of the same C/W ratio; however, by raising the sand-cement ratio of the former to 2.5 and keeping $C/W=2.26$, the depth of wear is decreased to about that for the latter. A similar comparison may be made between the 1:2.5 mortar and the 1:2:3 concrete for $C/W=2.05$. Here the replacement of one-half part of sand by three parts of ⅜-in. gravel decreases the depth of wear by almost 40 percent, though the cement content is halved. Thus, it appears that wear resistance depends both on proportion of aggre-

gate and on the C/W ratio, and that the drier the mix, within certain limits, the higher the resistance to wear.

That the decrease in depth of wear is not due merely to the presence of coarser material, such as gravel, is shown by the fact that a 1:2 mortar can be made dry enough so that the depth of wear will be equal to or less than that of the mixes containing gravel. For example, the 1:2 mix, $C/W=2.50$ (table 2, slabs 11 and 12) gives about the same total depth of wear as some mixes with gravel, such as slabs 46, 61, and 62. A still drier 1:2 mix, $C/W=2.82$ (slab 21) gave lower total depth of wear than any other, except those with special finishes, such as slabs 36 and 49.

For most of the mortar mixes the depths of wear in tests *A1* and *A2* were more nearly equal than for the mixes containing coarse

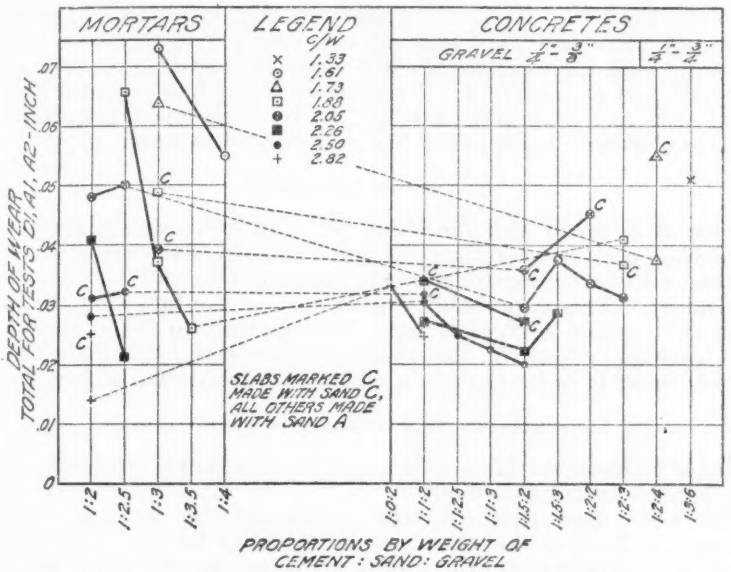

FIGURE 3.—*Depth of wear for mixes of various proportions and C/W ratios.*

All slabs trowled 4 hr or less after placing.

aggregate. For example, for slabs 75 to 78 and 80 to 85, which do not contain coarse aggregate, the *A2* wear was never less than 75 percent of the *A1* wear, the average value being 92 percent; for slabs 86 to 95, containing coarse aggregate, the wear in the *A2* test varied from 50 to 78 percent of the *A1* wear and averaged 61 percent.

Higher flow values were generally obtained with sand *C* for equal C/W ratios and mix proportions than with the finer sand, *A*. The effect on wear resistance of these sands is readily distinguished by use of the apparatus. The slabs with sand *C* generally showed greater wear for equal C/W ratios than those with sand *A*, as shown in figure 3. In some cases, however, there is little difference in either flow or wear, due to the use of different sands, as for the 1:1:2 mix, $C/W=2.50$ (slabs 46 and 86). The greater wear for some of the coarser sand

mixes is probably caused by difficulty in finishing, causing water to be brought to the surface as shown by slab 91 compared with slab 62, and slab 93 compared with slab 66. The mixes with gravel and the coarser sand require a higher proportion of sand for proper finishing, so that a 1:2:3 mix (slab 92) is preferable to a 1:2:4 mix (slab 93).

The mixes with gravel only, such as a No. 8 to ¼ in. or a ¼ to ⅜ in. showed comparatively little wear (slabs 22, 23, 29, 44). Slab 16, of 1:2 mortar, made with a sand all retained on the No. 16 sieve (sand *D*), showed very slight wear. With such an aggregate, the water content can be reduced to that corresponding to $C/W=2.82$. At the other extreme, a mortar with sand *E* all passing the No. 30 sieve showed fairly high wear.

For minimum wear it thus appears necessary to limit the amounts both of fine and of coarse aggregate as well as to limit the amount of water in the mix. High wear resistance may be secured by using a very dry 1:2 mortar, but it is more economical to replace a part of the sand with a greater weight of coarse aggregate. For instance, if one-half part of sand in a 1:2 mix, as in slab 12, is replaced by two parts of ¼- to ⅜-in. gravel (slab 58), the resulting mix is practically as easy to place and finish as the first and shows less wear.

(b) TYPE OF AGGREGATE

Five slabs made with limestone or "trap-rock" aggregate showed somewhat greater wear than the corresponding slabs made with the Potomac River aggregate; compare specimens 20 and 22, 55 and 57, 53 and 94. This is probably due in part to the fact that the slabs with the crushed aggregates were more difficult to finish because of the irregular shapes of the aggregate particles, or required more mixing water in order that they might be finished satisfactorily.

(c) FINISHING PROCEDURE AND DUST COATS

Table 2 shows the large differences in wear which were caused by varying the finishing methods.

The tests show that with few exceptions the depth of wear is reduced by leaving the surface undisturbed as long as possible (fig. 4) or by applying such mixtures as will cause the surface to be drier or to be richer in cement than the subsurface (fig. 5). It has already been shown that the drier mixes show the least wear. The greatest increase in wear resistance which can be brought about by delayed troweling is generally in that portion of the slab nearest the surface, as shown by results for the *D1* test, figure 4. A single delayed troweling appears to be more effective than two trowelings.

It is commonly believed that "fines," brought to the surface by troweling excessively or prematurely, are the cause of "dusting." The poor wear resistance of the wetter mixes appears more likely to be caused by water brought to the surface in finishing, since the addition of fines in the form of dust coats containing cement greatly reduces the depth of wear. The use of a dust coat also enables easier and earlier finishing in some cases, as for slab 68 compared with slab 67. The reductions in depth of wear effected by the use of dust coats are considerable in all three tests, as seen in figure 5. The dust coats, even when troweled into the surfaces immediately after placing, reduced the depth of wear; but greater reductions were obtained by

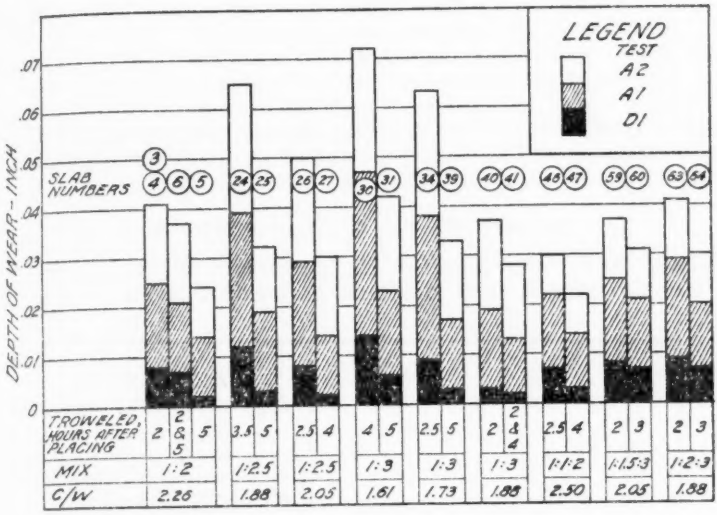

FIGURE 4.—*Effect of time interval between placing and troweling.*

Test *D1* = 5 min, no abrasive.
Test *A1* = 1st 10 min with abrasive
Test *A2* = 2d 10 min with abrasive.

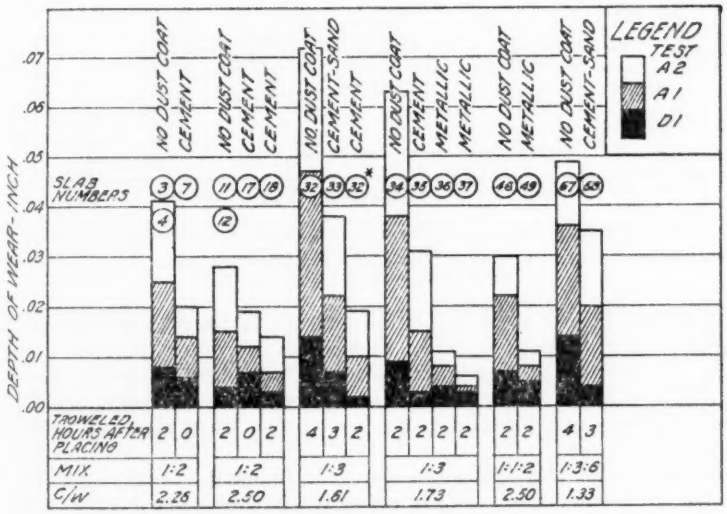

FIGURE 5.—*Effect of dust-coat finishes.*

Test *D1* = 5 min, no abrasive.
Test *A1* = 1st 10 min with abrasive.
Test *A2* = 2d 10 min with abrasive.
★1¼ lb cement per sq ft—slab badly crazed.

delayed troweling. (Compare slabs 11, 17, and 18, in table 2 and figure 5.) However, a large amount of cement dust coat, as on slab 32 (1¼ lb/in.^{2}) resulted in pronounced crazing.

Slab 37, coated with 0.9 lb of metallic aggregate mixture per square foot of surface, showed the least wear of all slabs tested.

Dust coats of sand alone (not reported in table 2) or of fine silicon carbide mixed with cement, used on slab 19, did not bond well and rubbed off easily. The same was true of the metallic aggregate containing 3 percent of oil by weight used on slab 38.

(d) CEMENTS AND CURING CONDITIONS

Slabs 69 to 77 were made for the purpose of comparing the effect of different cements and different curing periods on wear. The

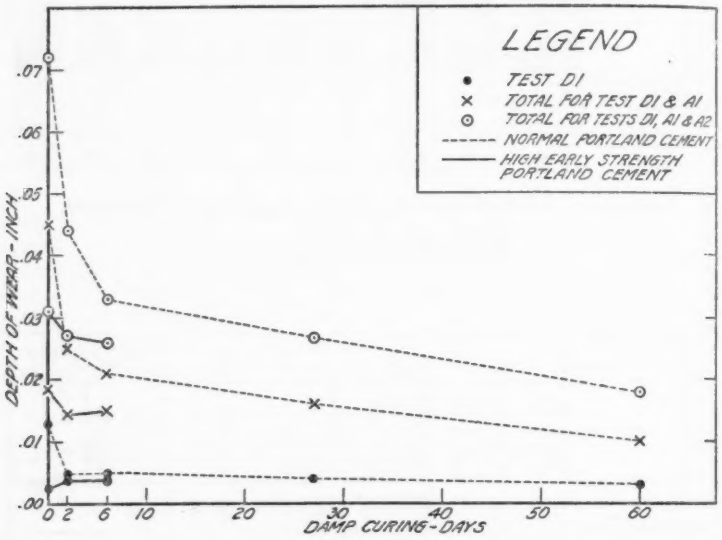

FIGURE 6.—*Depths of wear for different cements and various damp-curing periods.*

All slabs were made with a 1:2 mortar, C/W=2.50, troweled 2 hr after placing. For drying period after damp-curing, see table 2, Nos. 70 to 77.

previous tests had indicated that mortar is more suitable for this purpose than concrete because mortar shows comparatively greater wear. To minimize any effect due to troweling, a fairly dry mix (1:2, C/W=2.50), with sand *B*, was used in these tests. After the various damp-curing periods, the specimens were allowed to dry as indicated in table 2.

The results for this series are shown in figure 6. The values for the cement with the higher Fe_2O_3 content (No. 69) are rather close to those for the portland cement with less Fe_2O_3 and were therefore omitted for clarity of the figure. Without damp-curing, the slabs made with the "normal" cement showed low wear resistance. Damp-curing for only 2 days caused a considerable reduction in depth of wear—about 60 percent in the *D1* test, 40 percent in the *A1* test, 30 percent in the *A2* test, and about 40 percent for the total depth for all three tests. Continued damp-curing caused further reductions;

after 6 days the reduction in total depth of wear was about 55 percent and, after 60 days, about 75 percent.

The slabs made with the high-early-strength cement showed better wear resistance when not damp-cured than did the slabs with the "normal" cement damp-cured as much as 6 days. Although damp-curing improved the mortar made with the high-early-strength cement, such curing was not as necessary as for the slabs made with the "normal" cement.

Specimens made with the normal cement and not damp-cured (Nos. 13, 73, and 79) showed low wear resistance in the *D2* test compared with that in the *D1* test, as seen in table 2 for slabs 13 and 79. For damp-cured specimens the *D2* test generally indicated much higher wear resistance than the *D1* test, as for slabs 78, 84, 90, and 93.

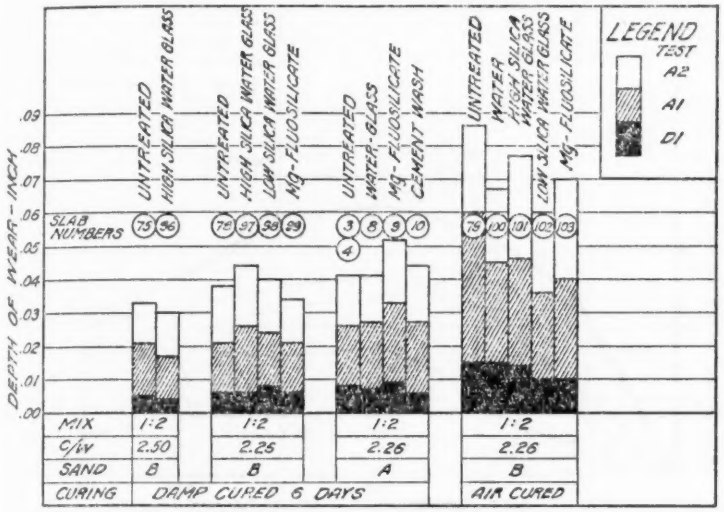

FIGURE 7.—*Effect of liquid surface treatments.*

All slabs were troweled 2 hr after placing. All surface treatments were applied when slabs were 28 days old.
Test *D1* = 5 min, no abrasive.
Test *A1* = 1st 10 min with abrasive.
Test *A2* = 2d 10 min with abrasive.

The wear resistance in the *D2* test was also poor for slab 30, made of wet 1:3 mortar. The *D1* test values gave an approximation of the extent of damp-curing under these test conditions when a normal cement was used. Thus, for slab 73, with no damp-curing, 64 g of dust was abraded in the *D1* test, whereas for the corresponding slab, 74, with 2 days damp-curing, only 27 g of dust was abraded.

(c) LIQUID SURFACE TREATMENTS

Eighteen slabs of 1:2 mortar were given liquid surface treatments. All of these slabs were duplicates of other slabs which had been tested without surface treatments. Ten of these were damp-cured for 6 days and eight were stored in the laboratory. All were treated at the age of 28 days.

Figure 7 shows the depths of wear for the treated slabs in the *D1*, *A1*, and *A2* tests in comparison with the depths of wear of untreated

slabs. The tests show that the liquid surface treatments did not produce a significant increase in wear resistance of damp-cured slabs but produced an improvement in slabs that had not been damp-cured. The application of water instead of the solution reduced the total depth of wear to some extent.

The series of half-minute tests, without abrasive, was made to determine the depths to which the surface treatments were effective. The results, given in figure 8, show that with three exceptions the greatest weight of dust was removed during the second or third half-minute test. The damp-cured specimens, as a group, showed less wear than those not damp-cured, and the slabs treated with magnesium fluosilicate showed the least wear in each group.

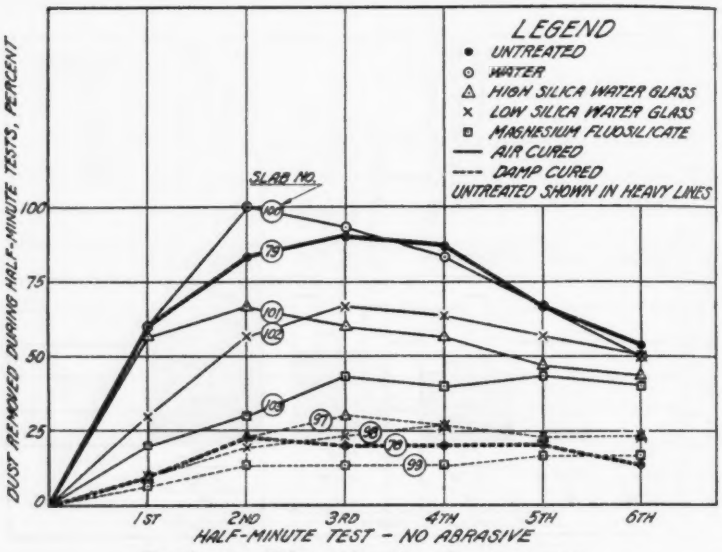

FIGURE 8.—*Effect of liquid surface treatments.*

All slabs were made with a 1:2 mortar, $C/W = 2.26$, troweled 2 hr after placing. The amounts of dust are expressed as percentages of the amount of dust removed from slab 100 during the second half-minute test (3.0 grams).

The tests show that in no case did surface treatments compensate for lack of damp-curing. The total depth of wear for all these half-minute tests was about 0.005 in. for the damp-cured slabs and 0.01 in. for those not damp-cured. At these depths the rates of wear in each group approached a constant value.

3. PITTING

The surfaces of the mortar slabs did not remain smooth after a small amount of wear had taken place; nearly all of them were moderately to excessively pitted. The various degrees of pitting are illustrated in figures 9, 10, and 11, which show portions of the worn surfaces. Because of the manner in which the photographs were taken, the pitting appears more pronounced than it would under ordinary illumination. A surface classed as excessively pitted would probably not be acceptable under any condition. "Moderate pitting" would ordinarily be acceptable except for especially high-grade work.

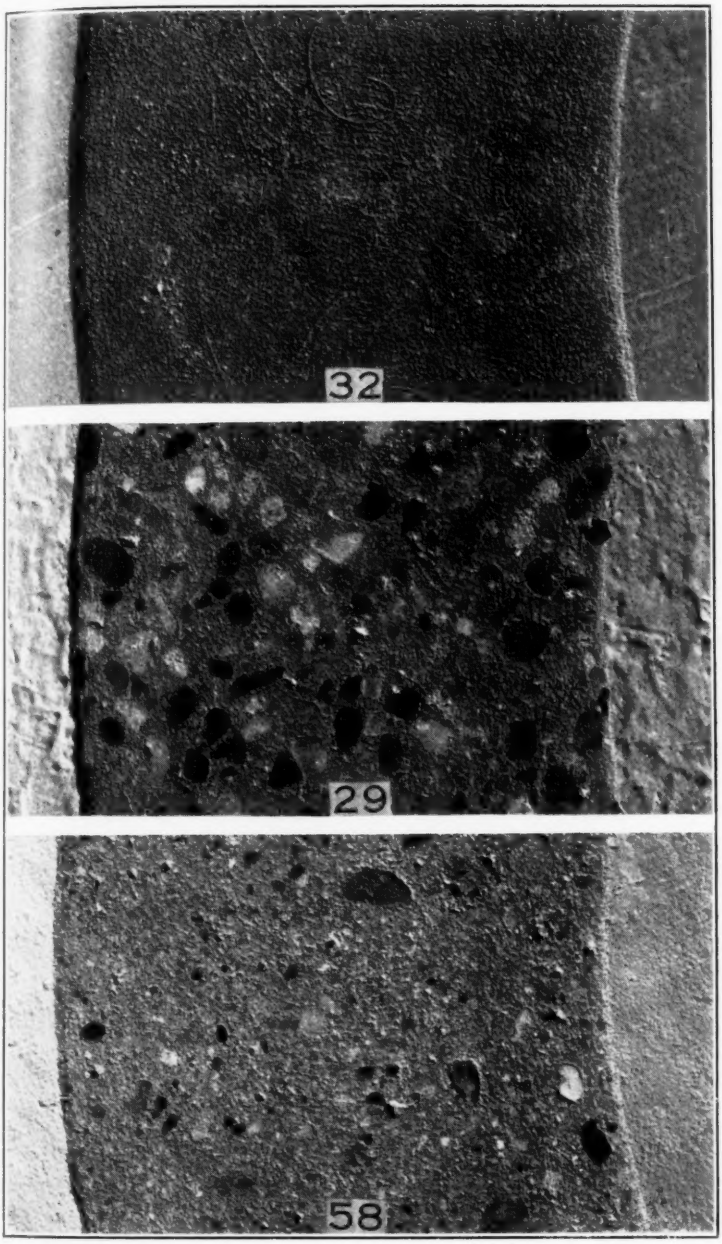

FIGURE 9.—*Appearance of slabs after test, showing degrees of pitting.*

Slab 32, negligible pitting; slabs 29 and 58, slight pitting.

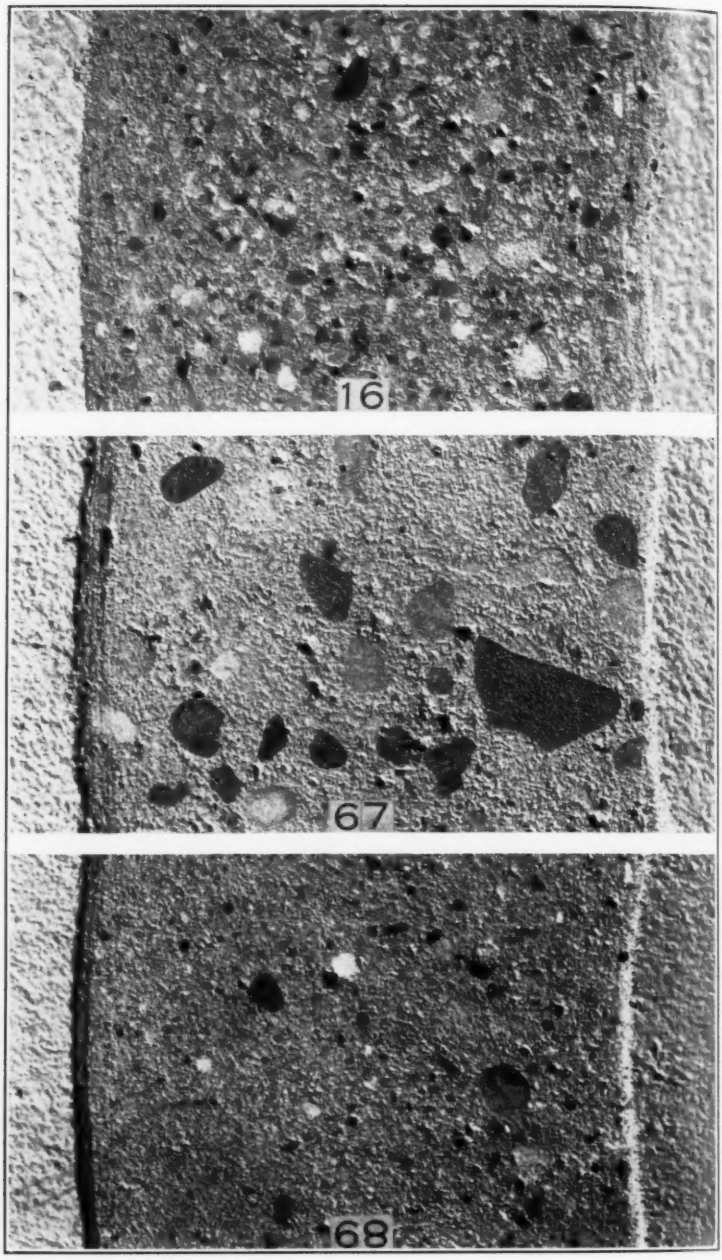

FIGURE 10.—*Appearance of slabs after test, showing degrees of pitting.*

Slabs 16, 67, and 68, slight pitting.

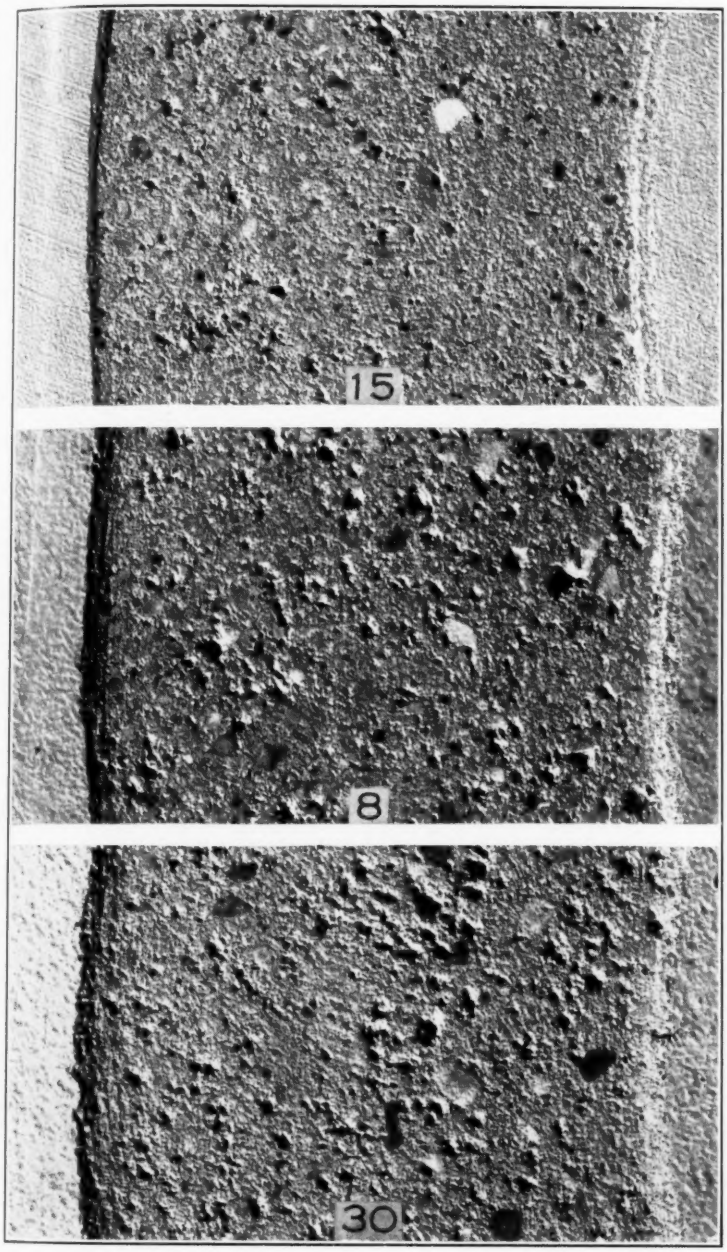

FIGURE 11.—*Appearance of slabs after test, showing degrees of pitting.*

Slab 15, moderate pitting; slabs 8 and 30, excessive pitting.

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Pitting may be due to exposure of voids in the mortar or concrete, or to actual loosening of particles of aggregate, as observed by Kessler [8] for some sandstones, in which the grains are comparatively hard but poorly bonded. The slabs in which the pitting after the *A2* test was slight were either dry 1:2 mortars or mixes with gravel in which the volume of mixing water was less than 24 percent of that of the concrete. Those slabs which were moderately pitted contained 24 to 28 percent by volume of mixing water. Those excessively pitted included the 1:3½ and 1:4 mortars, the mortars with water contents exceeding 28 percent, and mortars made with normal cement but not damp-cured—Nos. 13, 73, and 79. Generally the mixes which were badly pitted also showed comparatively poor wear resistance.

Dust coats reduced the degree of pitting, especially when applied to mortars; compare slabs 3 and 7; 11 and 17; 30, 32, and 33 (table 2). The only slabs in which the pitting was negligible were two to which dust coats had been applied (slab 32 with a dust coat of cement, and slab 37 with a metallic aggregate-cement dust coat). However, when the metallic aggregate contained oil (3 percent by weight), a slab (No. 38) to which it was applied showed excessive pitting.

VI. SUMMARY AND CONCLUSIONS

A portable testing apparatus for producing rapid abrasive wear of concrete floor surfaces and a device for measuring depths of wear were developed. The apparatus is placed directly on the surface to be tested and may be used with or without an added abrasive.

The test methods were applied in a study of concrete slabs 1 to 2 in. thick, by 23½ by 27½ in., and made of mixes varying from a 1:2 mortar to a 1:3:6 concrete. The *C/W* ratios, aggregate types and gradings, time intervals between placing and troweling, and curing conditions were varied. Surface treatments including liquid "hardeners" and metallic aggregates were also studied.

The dependability of the apparatus and method used for measuring the wear was determined by computing the standard deviations of the wear values. The computations showed that variations between the wear values of slabs made in duplicate were of the same magnitude as would be expected from the variations in eight individual measurements made on a single slab, indicating satisfactory reproducibility in slabs. The reproducibility of the test results as determined from the standard deviations indicated that the difference in wear values obtained for different slabs was significant and that the apparatus could distinguish the effect of variations in mixes, treatments, curing, and so forth.

A 5-minute test under the wearing action of steel disks, without an abrasive, shows large variations in wear resistance for various mixes, *C/W* ratios, time intervals between placing and troweling, and treatments such as dust coats or liquid surface treatments. The weight of dust removed in the test varied from 6 to 81 g, and the depth of wear from 0.002 to 0.018 in. The slabs showing the greatest wear in this test were those upon which excess water appeared on the surface during or after finishing.

After the 5-minute test, the resistance to wear of the subsurface was determined by two 10-minute tests with an added abrasive. The depths of wear during the first of these 10-minute tests varied from 0.001 to 0.045 in.

Nearly all of the slabs were more or less pitted after the smooth surface had been worn off. The mortars generally showed more pitting than the concretes.

The following conclusions may be drawn from the test results:

1. For the same mix proportions, the wear resistance of concretes and mortars is greater for higher C/W ratios. For equal C/W ratios, wear resistance increases with increasing proportion of aggregates up to a certain point, depending on the aggregates used, then decreases. Some mixes which had poor wear resistance at the surface were relatively more resistant after the surface "skin" was removed.

2. The shape of the aggregate particles (rounded or angular) affects the amount of water required for ease of placing and finishing, and thus may affect wear resistance of the concrete as much as the abrasion resistance of the particles themselves.

3. In this investigation, delaying the troweling for 3 or more hours after placing the concrete tended to increase wear resistance, especially for mixes containing no coarse aggregate.

4. The wear resistance of many mixes is greatly improved by troweling into the surfaces dust coats of cement, of cement and sand, or of cement and metallic aggregate. For example, the slab to which was applied a heavy dust coat consisting of a mixture of two parts of metallic aggregate to one of cement, by weight, had the highest wear resistance of all slabs tested. A dust coat also facilitates finishing the surface of concrete made of a lean mix, such as 1:3:6.

5. Concrete made with a "normal" portland cement and aged in the air without damp-curing may show comparatively poor wear resistance; but damp-curing may increase the wear resistance appreciably. Where a high-early-strength cement is used, the damp-curing is not as necessary.

6. Liquid surface treatments, such as solutions of magnesium fluosilicate or of water glass, are effective in improving the wear resistance of concrete that has not been damp-cured.

7. The use of coarse aggregates, such as gravel, permits reduction in the water content of the mixes and helps to reduce pitting. The use of dust coats containing cement also helps to reduce pitting, especially when applied to lean mixes.

VII. REFERENCES

[1] Report of Service Tests on Concrete Floor Treatments, Letter Circular NBS LC139 (Oct. 28, 1920).

[2] D. A. Abrams, *Wear tests of concrete*, Proc. Am. Soc. Testing Materials **21**, pt. 2, 13 (1921).

[3] C. E. Covell, *Heavy duty concrete floors*, Proc. Am. Concrete Inst. **27**, 454 (1928).

[4] Jackson and Pauls, *Accelerated wear tests of concrete pavements*, Proc. Am. Soc. Testing Materials **24**, pt. 2, 864.

[5] Ahlers, Linden, and Bird, *Wear tests on floor finishes at warehouse of R. H. Macy Co., Long Island, N. Y.*, Proc. Am. Concrete Inst. **24**, 778 (1929).

[6] J. G. Ahlers, *Good practice in concrete floor finish*, Proc. Am. Concrete Inst. **26**, 520 (1930).

[7] W. E. Hart, *Effect of materials and manipulation upon quality of finish*, Proc. Am. Concrete Inst. **26**, 532 (1930).

[8] D. W. Kessler, *Wear resistance of natural stone flooring*, BS J. Research **11**, 635 (1933) RP612.

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APPLICATION OF THE INTERFEROMETER TO THE MEASUREMENT OF DIMENSIONAL CHANGES IN RUBBER

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ABSTRACT

The interferometer method for the measurement of small dimensional changes is adapted here to measurements on soft rubber. A detailed description is given concerning the preparation of specimens, the apparatus, the procedure, and the calculations necessary for the application of this method. Particular emphasis is placed on the measurement of thermal expansivity and the dimensional changes involved in transitions between the different forms of rubber. The lower limit of temperature is set only by the refrigerating liquid, temperatures as low as −185° C being utilized. The upper limit of temperature is set only by the rubber itself. Results obtained in a study of the expansivity and transitions of "stark" (frozen) rubber are given as an illustration of the application of the method. Other possible applications are the measurement of the swelling of rubber by vapors and the determination of stress-strain relations under compressive loads. The chief limitation of the application of this method to rubber arises from the plastic flow of unvulcanized rubber at elevated temperatures. The method also possesses the inconvenience of requiring continuous observation. As compared with alternative methods of measuring small dimensional changes, the method possesses advantages arising from its high sensitivity, from the small size of the specimens required, from the ease of obtaining temperature equilibrium, from the absence of a confining liquid, and from the simplicity of the calculations.

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I. INTRODUCTION

The interferometer method for the measurement of small dimensional changes, previously used for many other materials, has been adapted in the present investigation to measurements on soft rubber. This method, which involves the counting of interference fringes of light of known wave length, was originated by Fizeau and developed by later workers. It has been most frequently applied to the measurement of thermal expansion. Merritt [1] has described the principles and the procedure which have been commonly followed at this Bureau in measurements of thermal expansion by the interference method. Measurements have been made on metals,[2] glass,[3] and ceramic materials,[4] especially at elevated temperatures.

The interference method has not been hitherto applied, however, as far as we are aware, to measurements on any of the various forms of rubber, with the single exception of ebonite, or hard rubber.[5] The relatively easy deformation of most kinds of rubber gives rise to problems which do not occur in the application of the interferometer to most other materials. It is the purpose of this paper to discuss the apparatus used and the technique employed in the measurements of dimensional changes in rubber and to give consideration to the advantages and limitations of the method in its application to rubber. Particular emphasis is laid on measurements at temperatures as low as that of liquid air for the determination of the expansivity and of the dimensional changes and temperatures involved in transitions between the different forms of rubber. The method is not limited to measurements at different temperatures, however. It may be used in any case in which the measurement of dimensional changes is required. Possible applications of this sort are the measurement of the swelling of rubber by vapors and the determination of stress-strain relations under compressive loads.

II. PREPARATION OF SPECIMENS

The specimens, which were placed between the two interferometer plates, usually had a thickness of about 3 or 4 mm. Two different types were utilized, the first a set of three small separate blocks [6] a few millimeters on an edge, and the second a ring about 21 mm in outside diameter and about 15 mm in inside diameter.

The small blocks were cut out by a device consisting of two single-edged safety-razor blades fastened on a steel bar, which was mounted by means of a hinge arrangement on a wooden base. The spacing of the razor blades, which were parallel and about 3.5 mm apart, determined the thickness of the specimens and made these thicknesses very nearly the same. No particular effort was made to obtain uniformity in the other two dimensions. After a considerable number of specimens had been cut from the same sample, three of them were inserted between the interferometer plates, and substitutions of other

[1] G. E. Merritt, *The interference method of measuring thermal expansion*, BS J. Research **10**, 59 (1933) RP515.
[2] C. G. Peters and H. S. Boyd, *Interference methods for standardizing and testing precision gage blocks*, BS Sci. Pap. **17**, 677 (1922) S436.
[3] A. Q. Tool, D. B. Lloyd, and G. E. Merritt, *Dimensional changes caused in glass by heating cycles*, J. Am. Ceram. Soc. **13**, 632 (1930); BS J. Research **5**, 627 (1930) RP219.
[4] G. E. Merritt, *Application of the interferometer to measurements of thermal dilatation of ceramic materials*, BS Sci. Pap. **19**, 357 (1924) S485.
[5] H. G. Dorsey, *Linear expansion at low temperatures*, Phys. Rev. **27**, 1 (1908).
[6] C. G. Peters, *The use of the interferometer in the measurement of small dilatations or differential dilatations*, J. Wash. Acad. Sci **9**, 281 (1919).

specimens were made, if necessary, until there were not more than 10 interference fringes visible across the face of the plate. In this manner, three blocks were selected which were of the same height to within a few wavelengths of light.

The ring specimens were cut with a die in an arbor press in the manner commonly used in the preparation of specimens for tensile tests. In this case, the height of the specimen, of course, was not changed by the cutting. Even from ribbed smoked sheet, it was found possible by cutting a large number of rings to select satisfactory specimens.

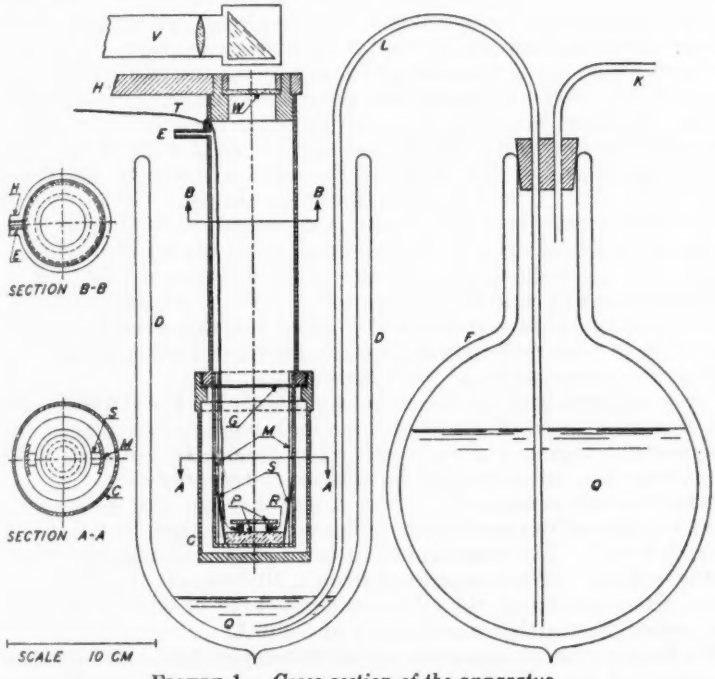

FIGURE 1.—*Cross section of the apparatus.*

C, removable part of the container; D, Dewar flask; E, metal tube; F, spherical Dewar flask; G, leather gasket; H, arm supporting container; K, glass tube; L, glass tube; M, strips holding support for interferometer plates; P, interferometer plates; Q, liquid air; R, rubber specimen; S, flat steel springs holding lower interferometer plate in place; T, thermocouple leads; V, viewing instrument; W, window.

III. APPARATUS

A diagram of the major part of the apparatus is shown in figure 1. It is essentially the same as that designed by Souder and Peters,[7] with modifications for work at low temperatures. The rubber specimen, R, separates two interferometer plates of clear, transparent fused quartz, the surfaces of each of which have been polished optically flat. The surfaces of the upper plate are inclined to each other at an angle of about 20 minutes in order to separate the reflections of the images from the upper and lower surfaces. A finely ground surface on the

[7] W. Souder and C. G. Peters, *An investigation of the physical properties of dental materials*, Tech. Pap. BS 13 (1920) T157.

bottom of the lower plate avoids undesirable reflection from this surface. The upper plate used in most of this work was 2.65 cm (1.05 in.) in diameter, 0.635 cm (0.250 in.) in thickness, and weighed 7.85 g. The lower plate is held in position on its support by two flat steel springs, *S*, one on each side. The springs are fastened to the two strips, *M*, which hold the support.

A bar, *H*, the height of which is adjustable by means of a rack and pinion not shown, supports the whole assembly. A removable part, *C*, of the container can be screwed to the stationary part, the joint being made tight by means of a leather gasket, *G*.

The temperature of the specimen is measured by means of a two-junction copper-constantan thermocouple placed between the interferometer plates and almost in contact with the specimen. The electromotive force of the thermocouple is read by means of a potentiometer. The leads, *T*, of the thermocouple pass through a small hole in the wall of the container near the top, the opening being sealed with wax. The reference junction is placed in a water bath in a small Dewar flask. The temperature of the bath is read on a mercury thermometer graduated to 0.1° C and changes only very slowly.

For measurements below room temperature, cooling is effected by means of liquid air. A Dewar flask, *D*, is placed around the container, *C*. A glass tube, *L*, extends to the bottom of this Dewar and to the bottom of a spherical Dewar, *F*, the liquid-air reservoir. Pressures above or below atmospheric applied through glass tube *K* make it possible to raise or lower the liquid-air level in Dewar *D*, and thus to control the temperature of the specimen.

For measurements above room temperature, the Dewars are removed, and a vessel of water or some high-boiling oil is placed around container *C*. This can then be heated by an electric hot plate in which the current can be controlled by slide-wire resistors.

Experimental observations are made through the glass window, *W*, at the top of the container. The viewing instrument, *V*, is of the Pulfrich type.[8] The source of light is an electric discharge tube containing helium. When operated from a 10,000-volt transformer, this tube emits a spectrum, the effective component of which is the prominent yellow line with a wavelength of 5876 A.

The height of each specimen at room temperature can be measured by means of some suitable instrument, such as the double-contact screw micrometer gage of the type designed earlier by Holt,[9] who has since improved the design of the electric-contact indicator by the substitution of a small neon lamp operated from the 110-volt line. The height of the specimen is obtained in this manner as the difference of readings taken on the interferometer plates, first with the specimen in position for measurements and then with the plates in contact with each other.

IV. PROCEDURE

The specimens must be placed in the apparatus in such a way as to make visible a relatively small number of interference fringes and to eliminate errors caused by the tilting of specimens. The condensation of moisture on various parts of the apparatus necessitates certain precautions as the temperature of the specimens is varied.

[8] C. Pulfrich, *The Abbe-Fizeau dilatometer*, Z. Instrumentenk. **13**, 365 (1893) **18**, 261 (1898).
[9] W. L. Holt, *Screw micrometer gages for rubber specimens*, BS J. Research **10**, 575 (1933) RP549.

1. PLACEMENT OF SPECIMENS

It was found desirable that both types of specimens, before being placed in the interferometer, have their upper and lower surfaces lubricated with finely powdered graphite, such as Acheson No. 2301. After a liberal application of the graphite the surface was rubbed on a sheet of paper to remove the excess of the lubricant. Direct observation with the screw micrometer gage showed that there was no appreciable change of the measured thickness occasioned by this operation. Lubrication of the surface was necessary, as is explained below, in order to avoid difficulties caused by the dimensional changes in the rubber in directions parallel to the interferometer plates.

After three block specimens of nearly equal height had been selected and lubricated as described, they were placed between the interferometer plates, near the edge of the upper plate and equidistant from each other. Next it was necessary to determine which of the specimens had the greatest height. This could be accomplished by pushing down gently on the center of the top interferometer plate, and noting the direction of motion of the fringes which is necessarily toward the point of greatest thickness.

The highest specimen was next moved toward the center of the plates, so that the load supported by that block could be increased from about one-third the weight of the upper plate to any desired value up to nearly the whole weight of the plate. The increased load on the highest block decreased the number of visible interference fringes, making the blocks, through the increased load, more nearly equal in height. Adjustment was made in this manner until not more than six or seven fringes appeared in the field of view.

It was next necessary to locate the point of contact carrying the greatest load. After a gentle tapping of the container supporting the plates, the interference fringe passing through the point of contact would remain stationary, while the other fringes would vibrate for a brief interval. The viewing instrument then was adjusted so that its cross hairs coincided with the point of contact.

A procedure essentially similar was followed when ring specimens were employed. Pressure applied on the top of the upper plate indicated which part of the specimen was highest. The ring was then moved so as to bring the high part closer to the center of the upper plate. The remainder of the procedure was the same as with the block specimens.

2. ELIMINATION OF ERRORS ARISING FROM TILTING

This method of making observations at the point of greatest load on the block specimens was suggested by J. B. Saunders, of this Bureau, who has recently discussed [10] the matter with particular reference to glass specimens. The method eliminated difficulties arising from the tilting of block specimens. Because of the difference in thermal expansivity between the specimens and the interferometer plates, there must be some relative motion of the surfaces of specimens and plates as the temperature is varied. If the surfaces are well lubricated, as by the use of graphite in the present work, this motion can take place as uniform slippage. If, however, there is sticking in

[10] J. B. Saunders, *Improved interferometric procedure with application to expansion measurements*, J. Research NBS **23**, 179 (1939) RP1227.

at least two specimens at points which are not immediately above each other, tilting of the specimens must occur, with consequent changes in the distance between interferometer plates. Such changes, of course, lead to false values for the expansivity, since the effective height of the specimen varies with the angle of tilt. An analysis of the stability of the system shows that a decrease in the angle of tilt at the specimen which is most heavily loaded, with consequently increased angles of tilt at the other specimens, results in a lowering of the center of gravity of the upper plate. Therefore, in actual practice all the tilting will occur at the lightly loaded specimens, and measurements at the point of contact of the most heavily loaded specimen will free the data from this error. Tilting of the lightly loaded specimens gives rise to a tilting of the upper interferometer plate. Such tilting, when it occurs, is evidenced by a change in the width of the interference fringes and consequently in the number of fringes visible. Thermal expansion alone causes a change in the width of the fringes of only the same percentage as the expansion itself. Since the expansion amounts to less than 3 percent over the whole range of temperature studied in the present work, and since changes of this amount in the width of the bands are almost unobservable, appreciable changes in the number of visible interference fringes may be ascribed entirely to sticking of the surfaces with consequent tilting.

Sticking of the surfaces of the ring and interferometer plates gives rise to deformations producing changes in the effective height of the ring, in a manner similar to the tilting of the block specimens. In this case, however, false values of the expansivity cannot be avoided by taking observations at the point of heaviest loading. It is, therefore, essential with ring specimens to avoid all sticking by the liberal application of lubricant. In some cases a tapping of the container was found necessary to bring about slippage.

3. ELIMINATION OF MOISTURE

Since the presence of moisture inside the apparatus gives rise to difficulties, the container should be closed tightly. The removable part, *C*, was consequently brought up around the specimens and screwed tightly in place. It was necessary, of course, during this process to avoid severe jolts, which might disturb the interference fringes. The joint was next wrapped with several turns of rubber tape, so that any water formed by condensation on the outside of the container might run down past the joint and not penetrate the leather gasket. The container was next evacuated through tube *E* and then filled with air which had been dried by passage over calcium chloride. If this were not done and if, consequently, water vapor in appreciable quantity were present inside the container, condensation would occur on the interferometer plates during warming from the lower temperatures, since the plates are then colder than the metal parts on which the vapor condenses during cooling. The presence of small amounts of condensed vapor on the plates can be tolerated, but beyond a certain limit light is no longer regularly reflected from the surfaces of the plates, and the field of view becomes dark.

If the humidity of the atmosphere was at all high, condensed moisture collected on the top surface of window W, when the container was kept at the lowest temperature. As a temporary expedient, the moisture was wiped off with a bit of absorbent cotton whenever present in sufficient quantities to interfere with vision; but it was found to be much more satisfactory to reduce the humidity of the room by an appropriate air-conditioning unit.

4. CONTROL OF TEMPERATURE

For measurements below room temperature, liquid air was used as the cooling medium. An atomizer bulb of rubber was placed on tube K and used to force the liquid air from the spherical Dewar, F, through tube L into Dewar D. By careful control of the level in D, the rate of cooling or warming of the specimen could be kept less than 1° C per minute, a value which it was found desirable not to exceed. The fringes passing the cross hairs were counted during both cooling and warming. The temperature was observed at the passage of every fifth fringe and on some occasions more frequently. The time also was recorded so that the rate of cooling or heating could be known. The temperature of the specimen, when the liquid-air level was brought to within a few inches of the top of Dewar D, became very nearly that of the liquid air, values lower than −180° C being always obtained after thermal equilibrium. After the minimum temperature had been attained, a considerable portion of the liquid air was removed from Dewar D by reducing the pressure in Dewar F by means of a pump connected to tube K. In this manner, the temperature of the specimen was allowed to rise at a controlled rate.

Before beginning observations at elevated temperatures, the bath of water or oil was cooled about 10 or 20 degrees below room temperature, and the current in the hot plate adjusted to a convenient value. It was found desirable to make as few changes in the heating current as possible, ranges of at least 20° or 30° being covered without change of the current. The rate of temperature rise decreased, of course, over each range. Preliminary runs were used to determine the value of the heating current for each range so that the initial rate of temperature rise should not exceed about 1° C per minute and that the final rate should not be unduly small.

V. CALCULATIONS

The essential data obtained during a run consist of the number of fringes which have passed the cross hairs, the corresponding voltage of the thermocouple, and the temperature of the fixed junction. From these one can calculate the change in length of the specimen and its temperature. Since the light traverses the space between interferometer plates twice, the vertical displacement of the upper plate when one fringe has passed the cross hairs is equal to one-half the wavelength. The change in length of the specimen between room temperature and any other temperature is obtained, therefore, by multiplying the number of fringes which have passed the cross hairs by one-half the wavelength. At room temperature the effective wavelength of the light used is 0.5876 μ. At other temperatures the wavelength is different because of the variation of the refractive

index of air with temperature. A table of corrections to take account [11] of this fact and the method of application are found in the paper by Merritt.[12]

The temperatures were read from a large calibration graph, which had been prepared from the standard tables for copper-constantan thermocouples given in the International Critical Tables.[13] The procedure described there was followed, a comparison between the thermocouple and a standard thermocouple being made at the temperature of liquid air.

A few selected lines of typical data and calculated results are shown in table 1 to illustrate the method usually followed. Columns 1, 2, 6, and 7 contain the quantities directly observed. The fourth column, "Air correction," contains values taken from Merritt's tables, for the correction for the change of refractive index of air with temperature. The seventh column gives the temperature of the fixed junction as read on the mercury thermometer. The next column gives the corresponding electromotive force obtained from the calibration curve of the thermocouple. The sum of this value and the potentiometer reading yields the potentiometer reading which would have been obtained had the fixed junction been held at 0° C, and this sum is shown in the ninth column.

TABLE 1.—*Typical data and calculations*

Original height of specimen, l_{20}=0.372 cm.

Wave length of helium line, λ=0.5876 μ.

Value of $\frac{\lambda}{2l_{20}}$=0.790 μ/cm.

Time	N fringe number [1]	0.790 N	Air correction	ΔL_1 expansion	Potentiometer reading	Temperature of fixed junction	Fixed junction electromotive force	Thermocouple corrected electromotive force	Temperature
		μ/cm	μ/cm	μ/cm	μV	°C	μV	μV	°C
9:00	−35	−27.6	0.1	−27.5	−1,310	23.6	1,900	590	7.4
9:02	−40	−31.6	.1	−31.5	−1,460	23.6	1,900	440	5.6
11:41	−283	−223.5	5.8	−217.7	−12,630	24.5	1,980	−10,640	−180.2
11:48	−283.7	−224.0	5.9	−218.1	−12,640	24.6	1,990	−10,650	−180.5

[1] N is the number of fringes which have passed the reference point, the negative value indicating contraction of the specimen.

VI. DIMENSIONAL CHANGES IN STARK RUBBER

The results obtained in a study of frozen rubber illustrate the type of work which can be done by this method. The form of frozen rubber investigated had a melting point above room temperature and has been called stark rubber by Pickles [14] and by Whitby.[15]

The change in dimensions resulting from the melting of a specimen of stark rubber is shown in figure 2. The sample, for which we are

[11] C. G. Peters and C. S. Cragoe, *Thermal dilatation of glass at high temperatures*, BS Sci. Pap. **16**, 449 (1920) S393.

[12] G. E. Merritt, *The interference method of measuring thermal expansion*, BS J. Research **10**, 59 (1933) RP515.

[13] L. H. Adams, Int. Crit. Tables **1**, 58. (McGraw-Hill Book Co., Inc., New York, N. Y., 1926).

[14] S. S. Pickles, *The consistency of rubber and rubber compounds*, India Rubber J. **67**, 69 (1924).

[15] G. S. Whitby, chapter 2, Physical Properties of Raw Rubber, in Davis and Blake: Chemistry and Technology of Rubber, p. 102 (Reinhold Publishing Corporation, New York, N. Y., 1937).

indebted to H. I. Cramer, of the University of Akron, had been in the frozen state for less than a year. The initial heating rate was approximately 0.4° C per minute and decreased to about 0.16° C per minute at the highest temperature. At this point the heating bath was removed and the specimen allowed to cool. The temperature decreased at an initial rate of about 0.95° C per minute, and the rate diminished to less than 0.1° C per minute at the last observation.

The difference in ordinates of the heating and cooling curves at 25° C represents the difference in heights of the amorphous and the crystalline forms. It can be read from the graph as 91.5 μ/cm. Assuming the specimen isotropic, one finds that this corresponds to a volume increase of 2.77 percent, a value not very different from those found [16] at 0° C for rubber which melted near 11° C. The general form of the graph is typical of the melting curve of rubber, but linear expansivities calculated from the slopes in figure 2 are abnormally high, probably because the specimens were not lubricated during this

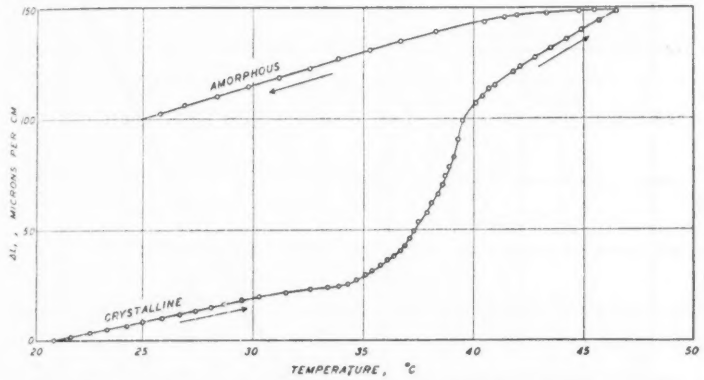

FIGURE 2.—*Dimensional changes during the melting of stark rubber.*

particular run. The importance of lubrication was not appreciated at the time of this measurement.

The results of measurements of the dimensional changes in a different sample of stark rubber over a large range of temperatures are shown in figure 3. We are indebted to G. S. Whitby for furnishing the sample, which had been in the frozen state for 25 years (from 1913 to 1938).

The specimens were cooled from room temperature to that of liquid air. They were allowed to warm up nearly to room temperature, and electric heating was then applied. The melting range in this case began at about 40° C, but observations could not be continued above 43° C, because at this point permanent deformation of the specimens under the weight of the upper plate occurred.

In figure 3 the circles represent experimental points obtained when the temperature was changing at a rate of less than 1° C per minute. Under these conditions it is thought that the difference between the actual effective temperature of the specimens and that indicated by

[16] N. Bekkedahl, *Forms of rubber as indicated by temperature-volume relationship*, J. Research NBS **13**, 411 (1934) RP717.

the thermocouple is negligible. This is shown by the good agreement between the values obtained as the temperature was increasing (dark circles) and the values obtained as the temperature was decreasing (open circles). Observations made when the temperature was changing at a rate greater than 1° C per minute are shown as dots. Under these conditions a difference between values obtained with increasing and decreasing temperatures can be seen.

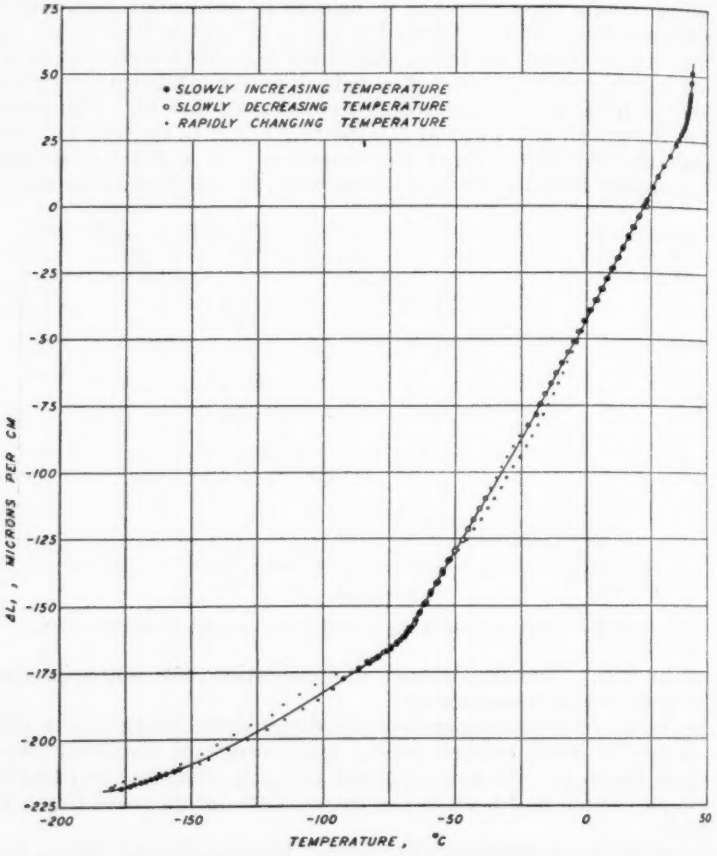

FIGURE 3.—*Dimensional changes in stark rubber.*

The same data in the interesting region from −54° to −84° C have been plotted on a much larger scale in figure 4 to show the transition[17] of the second order near −70° C. The linear expansivity from −71° to −84° C is read from the graph as 77×10^{-6} per degree, while the linear expansivity from −67° to −54° C is found to be 175×10^{-6} per degree. These ranges of temperatures are, of course, insufficient to show the continuous variation of expansivity with temperature. The variation is evident over the larger ranges shown in figure 3.

[17] N. Bekkedahl, *Forms of rubber as indicated by temperature-volume relationship*, J. Research NBS 13, 411 (1934) RP717.

VII. ADVANTAGES AND LIMITATIONS OF THE METHOD

The interference method of measuring dimensional changes in rubber possesses certain advantages as compared with other methods, and, of course, certain attendant disadvantages. There are, in addition, certain limitations which restrict its use. The advantages and limitations will now be compared with those of the chief alternative methods.

1. DIMENSIONAL MEASUREMENTS

If there is reason to think that the rubber specimens are not isotropic, some method, such as the interference method, by which one can measure separately the three linear dimensional changes, is required. Any method involving merely the measurement of volume changes would give insufficiently detailed information about the changes in different directions.

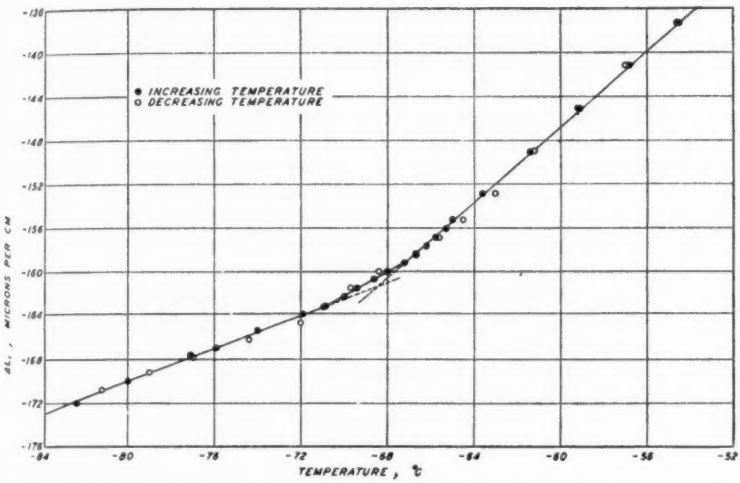

FIGURE 4.—*Dimensional changes in stark rubber passing through a second-order transition.*

This is a part of figure 3 shown on a larger scale.

2. SIZE AND SHAPE OF SPECIMENS

The interference method has been found more satisfactory for rubber than other familiar methods for linear measurements, which require a specimen in the form of a rod and which measure expansions by means of a dial gage,[18] [19] optical lever, or similar arrangement.

The interferometer specimens are very small and can easily be cut to shape. In spite of the small size of the specimens, the sensitivity of the method is quite great, since measurements of changes of length are made by comparison with the wave length of light. In the other methods the specimens need to be much larger and must be formed or molded to a particular shape.

[18] P. Hidnert and W. T. Sweeney, *Thermal expansion of magnesium and some of its alloys*, BS J. Research 1, 771 (1928) RP29.

[19] N. Bekkedahl, *Forms of rubber as indicated by temperature-volume relationship*, J. Research NBS 13, 411 (1934) RP717.

3. FORCE ON SPECIMENS

Most of the methods used for linear measurements exert some constraint on the rubber, either from the measuring mechanism or from the means of support. In the case of the interference method, the lateral constraint from the support is made quite small by the lubrication with graphite, but the vertical constraining force on the specimen due to the weight of the upper interferometer plate is, of course, of appreciable magnitude. This force is constant for a given plate and is of known size. Moreover, by the use of different plates, the force may be altered. Satisfactory plates weighing considerably less than 1 g can be made.

4. PLASTIC FLOW

Plastic flow of the unvulcanized rubber was not observed at the lower temperatures and did not occur until temperatures somewhat above 40° C were reached. The temperatures at which the plastic flow began varied somewhat from sample to sample but could be located in each case within at least half a degree. Plastic flow made interference measurements impossible on unvulcanized rubber at higher temperatures. With vulcanized rubber no temperature limit set by plastic flow was observed in a series of measurements which extended to 100° C. No type of linear-expansion measurement can be free from the limitation caused by plastic flow, when it occurs. However small the constraining forces, this effect will occur at the higher temperatures even under the weight of the specimen itself.

If the rubber is isotropic, methods which measure volume change can be utilized and are necessary in cases where there is a possibility of plastic flow. A dilatometer with confining liquid has been used [20] to measure volume changes in rubber and is probably the most important alternative to the interference method. Here plastic flow is greatly reduced and, even when it occurs, does not alter the volume. The method can thus be used for unvulcanized rubber at higher temperatures.

5. CONFINING LIQUIDS

The interference method requires no confining liquid in contact with the rubber and is free from difficulties arising from the presence of the confining liquid usually necessary in a volume dilatometer.

None of the common liquids which are thought to be without swelling action on rubber remain liquid much below −100° C, and, in actual practice, work using a confining liquid has been limited to the range above −85° C because of the excessive viscosity of those liquids which do not actually crystallize above −100° C. The interference method is free from this temperature limitation, and measurements have been continued to temperatures as low as that of liquid air. With certain modifications of technique, the temperatures might be carried still lower to limits set only by the refrigerating liquid.

Furthermore, it has not been established that the presence of a confining liquid, even one which does not swell rubber appreciably, is completely without influence on the occurrence of transitions. For example, there seems to be evidence [20] that the presence of acetone

[20] N. Bekkedahl, *Forms of rubber as indicated by temperature-volume relationship*, J. Research NBS 13, 411 (1934) RP717.

inhibits the second-order transition normally occurring near −70° C, or at least lowers the temperature at which it occurs.

Other disadvantages of a confining liquid are the constant possibility of evaporation during a run, and the difficulties caused by the liberation of dissolved gases, as the temperature is raised. Usually, if a fair degree of sensitivity is required, ranges of not more than 30° or 40° C can be covered without the addition or removal of liquid.

6. TEMPERATURE EQUILIBRIUM

In comparison with other methods for either linear measurements or volume measurements, the specimens used in the interferometer are quite small. Therefore, in addition to the resultant economy of material, there is the advantage that the attainment of temperature equilibrium throughout the rubber is relatively rapid. Consequently, changes of temperature can safely be made much more rapidly than in cases where large specimens are used, when the attainment of equilibrium is delayed by the relatively poor thermal conductivity of rubber. The expansivity over the range of temperatures from that of liquid air to room temperature can therefore be measured by the interference method in a single working day. With some of the other methods, observations must extend over several days. For dimensional changes of such nature as to require more than 1 day for completion, such as might occur during swelling and freezing, the interference method is not so convenient, since it requires continuous observation.

This inconvenience might be removed by adopting photographic recording [21] or a different interference method involving the use of a block of fixed height and the counting of the number of interference fringes between two marks rather than the counting of the number of fringes which pass a given mark.

Because of the relatively rapid attainment of temperature equilibrium in the interference method, it is convenient to carry out measurements with decreasing temperatures as well as with increasing temperatures. Thus, any permanent distortion is immediately obvious.

7. CALCULATIONS

The calculation of expansivities from interference measurements is considerably simpler than from volume measurements. In the latter case, for example, it is necessary to take account of the expansion of the glass bulb and of the confining liquid, as well as to make corrections for the temperature of the exposed stem. The different amounts of confining liquid necessary for different temperature ranges further complicate the calculations.

The interference method introduces no discontinuous intervals of temperature into the calculations. As shown by figures 2 and 4, it can give results of considerable detail in studies of the nature of a transition.

WASHINGTON, September 1, 1939.

[21] F. C. Nix, *A vacuum interferometric dilatometer with photographic recording*, Phys. Rev. 55, 606A (1939);

U. S. Department of Commerce National Bureau of Standards

RESEARCH PAPER RP1254

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COSMIC-RAY OBSERVATIONS IN THE STRATOSPHERE WITH HIGH-SPEED COUNTERS

By Leon F. Curtiss, Allen V. Astin, Leroy L. Stockmann, and Burrell W. Brown

ABSTRACT

By a modification of a method[1] reported previously, observations of cosmic-ray intensity at high altitudes have been made.

A Neher-Harper circuit has been used with relatively large Geiger-Müller counters so that 2,000 to 3,000 counts per minute could be recorded by means of radio equipment sent aloft attached to free balloons. The counters were standardized in terms of the radiation from 1 mg of radium at a distance of 1 m, and the maximum cosmic-ray intensity was found to be 0.55 percent of this intensity. This maximum was found to occur at an atmospheric pressure of 60 millibars. A very low intensity at pressures of about 5 millibars, reported in the previous paper, was not observed in any of the 15 ascents in which the data reported in the present paper were obtained.

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I. INTRODUCTION

In a previous paper[2] results were given of cosmic-ray observations in the stratosphere, using small, low-speed Geiger-Müller counters. The counters were carried aloft by sounding balloons, and the counts were broadcast to a receiving station on the ground by a radio transmitter also carried by the balloons. The active volume of each counter was approximately 1 cm^3, and the counting rate at the altitude of maximum intensity was approximately 60 per minute. Since the counting rate on the ground was less than one per minute and since the time required for the equipment to reach the altitude of maximum counting rate was only 90 minutes, the results were subject to considerable statistical fluctuation.

[1] Phys. Rev. **53**, 23 (1938).
[2] L. F. Curtiss, A. V. Astin, L. L. Stockmann, B. W. Brown, and S. A. Korff, Phys. Rev. **53**, 23 (1938).

In the present investigation the size of the counters has been increased to give approximately a thirtyfold gain in counting rate. This increase was sufficient to make the curves of counting rate versus pressure comparable in smoothness to those obtained from observations with ionization chambers. The great advantage of the counter method over the ionization chamber for cosmic-ray exploration of the upper atmosphere is that the balloons do not have to be recovered in order to obtain the data.

A further improvement in the present investigation over earlier work has been the standardization of each counter in terms of gamma radiation from radium.

II. DESCRIPTION OF BALLOON EQUIPMENT

1. COUNTERS

The general construction of the Geiger-Müller counter tubes used in this investigation may be seen in figure 1, which shows a typical tube. The outer electrode, or cathode, is a copper tube about 1 cm in diameter and 16 cm in length. The anode is a No. 42 steel wire with glass sleeves at the ends to serve as guards, after the manner described by Cosyns and de Bruyn.[3]

To secure a broad and level plateau, it has been found necessary to clean the electrodes by sputtering. The treatment employed is a modification of the method described by Diffenbach, Lifshutz, and Slawsky.[4]

The electrodes are connected to a transformer adjusted to produce a steady glow between the electrodes in an atmosphere of hydrogen at a pressure equivalent to a few millimeters of Hg. The hydrogen is changed frequently and the sputtering permitted to continue for from 12 to 24 hours. Care must be taken during the sputtering to prevent a deposition of metal on the glass surface so that the insulation may not be ruined.

After sputtering, the counters are filled and sealed with a mixture of about 10 parts of argon to 1 part of hydrogen at a pressure of approximately 6 cm of Hg. The counters so treated and filled require about 575 volts for their operation and have an approximately constant counting rate for any voltage, either higher or lower, within 50 volts of this value.

2. INTEGRATING CIRCUIT

The wiring diagram of the amplifier and integrating circuit associated with the counter is shown in figure 2. The pulses from the counter are fed to a high-speed stage of the Neher-Harper [5] type.

The major purpose of this stage is to restore rapidly the counter to its normal state. The second stage serves to level the pulses, since those coming from the first stage may have widely different magnitudes. The tube is arranged for a sharp cut-off, so that all input pulses, regardless of their magnitude, give the same output signal. The third stage rectifies the pulses and the rectified current charges the capacitor, $C4$. When the charge on $C4$ is sufficient to flash the neon tube, N, a signal is transmitted through the final stage, actuating

[3] Acad. roy. Belg., Bul. classe sci. **20**, 271 (1934).
[4] Phys. Rev. **52**, 1231 (1937).
[5] Phys. Rev. **49**, 940 (1936).

FIGURE 1.—*Geiger-Müller tube counter used with radio-balloon equipment.*

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the relay, *Z*. The relay is arranged to key the radio transmitter. The flashing voltage of the neon tube and the size of capacitor *C4*, as well as the magnitude of the leveled pulses, determine the number of counts or discharges in the Geiger-Müller tube necessary to actuate the relay. This number has varied from 35 to 100 in the 15 units that have been used for stratosphere soundings.

The integrating circuit, which is similar in some respects to one described by Johnson,[6] is essential to prevent a large increase in the keying rate of the transmitter when the counting rate is stepped up. This is because of speed limitations of the ground recording system. In the earlier work of the authors with low-speed counters the integrating circuit was not essential. It was found that with the amplifying and integrating circuit of figure 2, higher counting rates could be handled than could be provided with counters of the size described

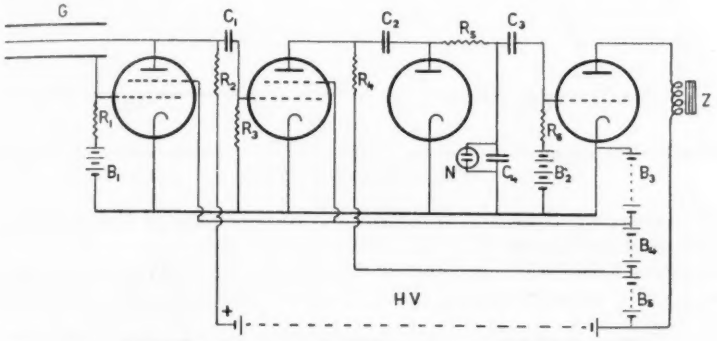

FIGURE 2.—*Wiring diagram of integrating circuit used with tube counters.*

G=tube counter	R_1=20 megohms	*N*=neon lamp
C_1=0.0001 μf	R_2=5 megohms	B_1=6 volts
C_2=0.0001 μf	R_3=1 megohm	B_2=12 volts
C_3=0.01 μf	R_4=1 megohm	B_3=22.5 volts
C_4=0.01 μf.	R_5=20 megohms	B_4=90 volts
	R_6=10 megohms	B_5=135 volts

in the previous section. Accordingly two counters were connected in parallel, thus approximately doubling the counting rate.

The characteristic of a typical counter and its associated circuit as a function of voltage applied to the counter is shown by figure 3. The fact that the counting rate is constant over a 95-volt range shows that not only does the counter have a satisfactory plateau but that the leveling stage of the amplifier is operating satisfactorily. Tests of the linearity of the integrating circuit have been made in three different ways with equally satisfactory results in each case. With different intensities of radiation applied to the counter, the flashing rates have been compared to the counting rates of a Cenco counter connected directly to the output of the first stage of the amplifier so as to record the impulses of the counter directly. The number of impulses per flash was the same for all counting rates tested. The second test consisted in placing radium samples of different known intensities at a fixed distance from the counter tube. The ratios of the differences in counting rates were the same as ratios of the differences in

[6] Phys. Rev. **53**, 914 (1938).

strengths of radium sources. The third test involved a fixed quantity of radium at different distances from the counter. The ratios of the differences in counting rates coincided with the expected differences from inverse-square law calculations.

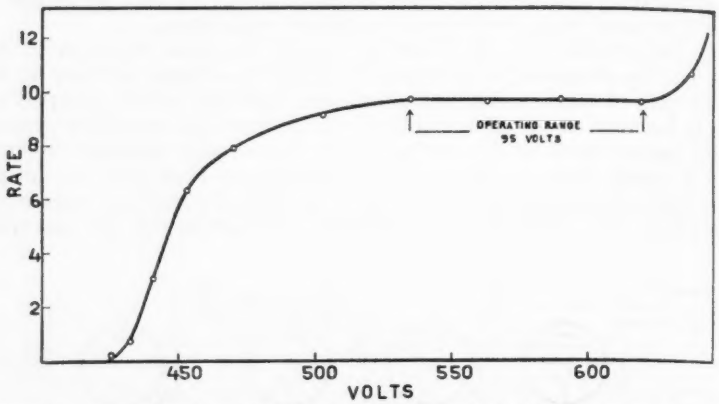

FIGURE 3.—*Operating characteristic of typical counter showing variation of counting rate with applied voltage.*

The resolving time and maximum counting rate of the circuit were determined by the method suggested by Schiff.[7]

In figure 4 the solid line shows Schiff's theoretical curve of counting rate, (N/T), times resolving time, (τ), plotted against the ratio of total

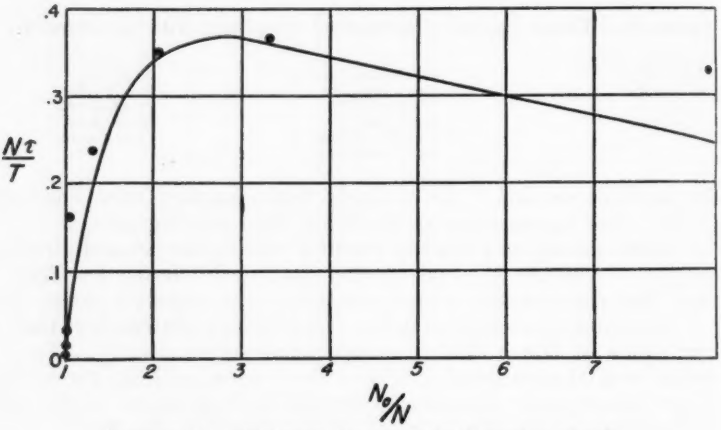

FIGURE 4.—*Efficiency characteristic of counter circuit.*

N=Total number of pulses counted in time T.
N_0=Total number of pulses in counter in time T.
τ=Resolving time of circuit.

particles through the counter, (N_0), to the number recorded by the counter, N. The maximum counting rate occurs when $N_0/N=e$ and the resolving time is determined from the value of the maximum, $N\tau T/T=1/e$. The circles show the experimental points which were

[7] Phys. Rev. 50, 88 (1936).

FIGURE 5.—*Complete assembly including counter tubes, vacuum-tube circuit, barograph, radio transmitter, and necessary batteries.*

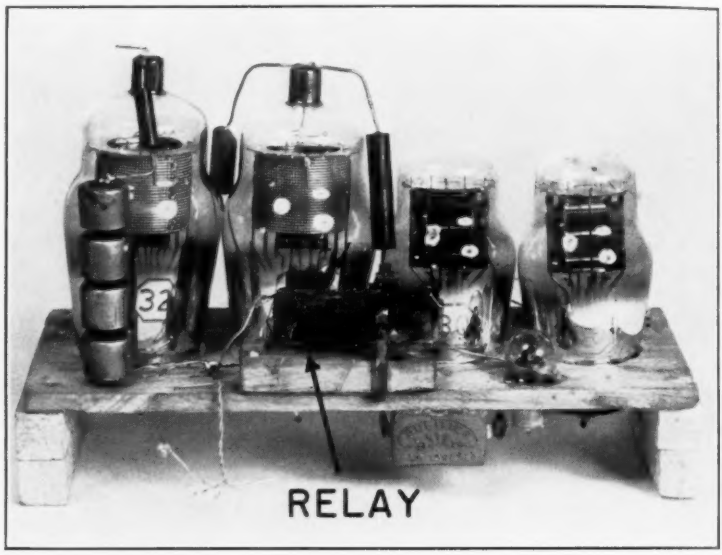

FIGURE 6.—*Amplifying and integrating assembly.*

obtained by observing the counting rates when radium sources of different known intensities were placed at a fixed distance from the counter. The values of N_0 were fixed from the lower counting rates where a linear relation was observed between counting rate and intensity of the source (after allowing for the blank counting rate). Theory and experiment show the maximum counting rate at the same value of N_0/N, but differ in other respects, as shown in the figure. These differences might occur if the resolving time, τ, were not constant, as was assumed by Schiff. The value obtained for τ from this set of data was 9×10^{-4} second. In the observations on cosmic rays the counting rates never exceeded a value for $N\tau/T$ of 0.05, for which values N_0/N was unity, within a few percent.

3. BAROGRAPH

The barograph used in this investigation was of the same type as that used in the earlier work and has been described in detail elsewhere.[8] The sensitive element of the barograph was a small aneroid capsule of copper-beryllium alloy. The revolving contact arm of the barograph was driven by a small impulse-type motor which was adequately powered by a 1½-volt "pen-light" battery. Pressures could be determined to within ±2 millibars over the range of pressures 1,000 to 5 millibars. The possible error due to temperature variations of the barograph has been carefully investigated and found to be less than the precision of reading over the entire range.

4. RADIO TRANSMITTER

The radio transmitter was also the same as that used in the earlier work. It consisted of two type 30 tubes in push-pull oscillating at 55.5 megacycles and powered by a 135-volt plate supply. Keying was accomplished by short-circuiting a cathode biasing resistor. The resistor was sufficiently large so that the circuit would not oscillate unless the resistor were shorted. Consequently, the transmitter was silent except for the relatively short times of keying, an important factor in keeping the battery requirements at a minimum.

5. BATTERIES

The voltage supply for the counter (*B5*, fig. 2) was obtained from Eveready X–180 units. Each one of these units supplies 45 volts and weighs but 2 ounces. The plate voltage for the tubes of the amplifier, as well as for the transmitter, was obtained from Burgess V40 Fl units. Each of these units furnishes 45 volts and weighs about 3.5 ounces. The filaments of all tubes required 2 volts for their operation, which was obtained from ordinary flashlight batteries. It was found most convenient to connect two filaments in series and heat them with the current from three flashlight batteries in series.

6. ASSEMBLY

The assembled balloon equipment is shown in figure 5. The four tubes of the amplifier and integrating circuit, a separate view of which is shown in figure 6, are in the center of the picture. The plate-supply

[8] Phys. Rev. **53**, 23 (1938); J. Research NBS **22**, 97 (1939) RP1169.

batteries for these tubes, as well as for the transmitter, are just behind them. In front of the amplifier are the neon tube and relay (*Z*, fig. 2) for keying the transmitter. The second counter has been removed but is normally installed just in front of the amplifier. Below the amplifier may be seen the high voltage supply for the counters. To the right of the amplifier is the barograph unit, and to the left is the radio transmitter. The antenna and attachment cord to the balloons are shown in the upper part of the picture.

The crate in which the equipment is mounted consists of a balsa-wood frame with double Cellophane lining for heat insulation. The front and back sides of the crate are removed in figure 5. The interior Cellophane surface is lined with black paper, which serves both as a heat absorbing surface and as a light shield for the counters. Tests on the interior temperatures of such enclosures when in the stratosphere in daytime have shown the temperature to be within $\pm 15°$ C of 25° C.

It was found that moisture enclosed within the frame when the crate was sealed frequently condensed on parts of the insulation and interfered with the proper operation of the equipment. This trouble was eliminated by coating the insulating surfaces well with Superla wax and enclosing some calcium chloride in a cheesecloth bag within the crate when it was sealed.

The weight of the completely assembled balloon equipment ready for attaching to the balloons was approximately 2,200 g.

7. BALLOONS

The counter equipment and radio transmitter were carried aloft by five or six Dewey and Almy latex balloons, model No. 700. These balloons have an uninflated diameter of about 4 ft and will expand to approximately 19 ft before bursting. The balloons were inflated with hydrogen to have a free lift of approximately 600 g per balloon. This gave an ascension rate in excess of that normally used in stratosphere soundings, but it has been the authors' experience that higher altitudes are obtained with higher ascensional rates.

The balloons were attached to strings of different lengths to reduce contact of the balloons with each other. This was found desirable in preventing both premature and simultaneous bursting of the balloons.

III. GROUND EQUIPMENT

The receiver used in this work was a commercial 5-meter super-regenerative type. A detector tube of the grid current type was added to the receiver. In its unexcited condition the regenerative noise of the receiver suppressed the plate current of the detector tube. A signal entering the receiver suppressed the regenerative noise and caused an increase in the current of the detector tube. The receiver was very insensitive to noise, since only a definite pulse of waves of the frequency to which the receiver was tuned would suppress the regenerative noise and thus release the output current. Ordinary static was quite ineffective, since it mainly added to the regenerative noise. The disadvantage of the circuit was that a signal of at least about one-tenth second duration was necessary to actuate the receiver. This, of course, limits the number of signals per second which the

receiver can handle and explains why it was essential to use an integrating circuit on the balloon equipment for high-speed counters.

The method of actuating the recorder is shown in figure 7. The plate current from the detector tube enters at the terminals marked "to amplifier" and passes to the relay. A signal in the receiver increases the current through the meter, M, and actuates the relay, which in turn controls the grid potential of the gas-filled 885 triode. The plate current of the 885 tube passes through an electromagnet which controls a recording stylus.

The complete ground equipment is shown in figure 8. The receiver is to the right; the plate-current meter of the detector circuit, the 885 tube, and the power supply are in the center; and the waxed-tape record and recording stylus to the left. Perforated tape, moved by a sprocket which is driven by a synchronous motor, is used to secure uniform motion of the tape.

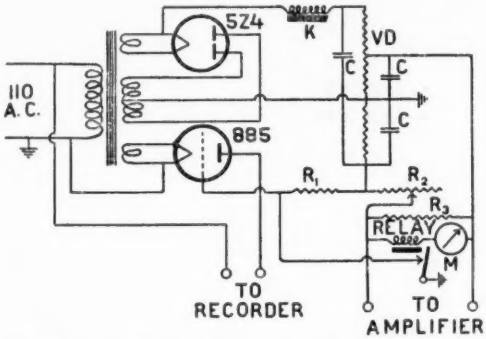

FIGURE 7.—*Wiring diagram of detector circuit used with superregenerative receiver to actuate records.*

VD=25,000 ohm voltage divider
K=30 henry choke
C=8 μf
R_1=0.5 megohm
R_2=50,000 ohms
R_3=100,000 ohms

A sample record is shown in figure 9. The upward breaks in the horizontal line on the tape correspond to signals received from the radio transmitter. The double breaks marked "reference doublet" correspond to regular keyings of the transmitter by the motor-driven contact arm. These reference marks are received approximately four times per minute. The break marked "pressure" corresponds to a keying of the contact arm by a contact point connected to the aneroid capsule. The ratio of the distance between a reference doublet and pressure signal to the distance between two reference doublets gives a measure of the pressure. The narrow breaks in the horizontal line marked "cosmic rays" correspond to keyings of the transmitter by the integrating circuit. These signals are readily discernible from the barograph signals by both their width and position. The regularity of the spacing between the cosmic-ray signals is an indication of the extent to which the random fluctuations in the individual discharges of the counter are smoothed out by the integrating circuit.

IV. RESULTS

A total of 15 successful sets of observations were obtained with equipment of the type just described, between July and November 1938. A typical record, which was obtained July 22, is shown in figure 10. The record shows excellent agreement in results obtained during both the ascent and descent of the balloons. The ordinate scale shows the actual number of discharges per minute in the counters, which for this particular equipment was 48 times the flashing rate of the neon tube in the integrating circuit, or 48 times the number of cosmic-ray radio signals received at the ground station. The number of counts required to flash the neon tube was determined by means of a Cenco counter.

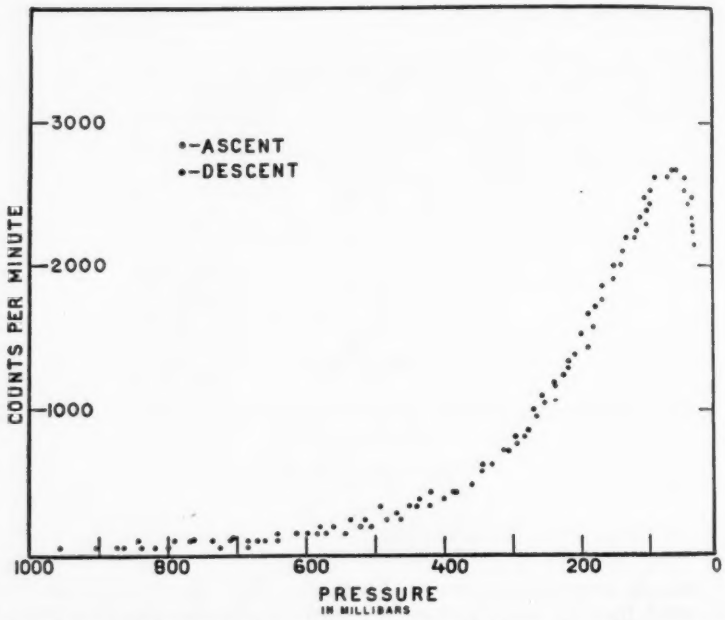

FIGURE 10.—*Data obtained on a single ascent and descent, showing counts per minute plotted against the atmospheric pressure in millibars.*

A summary of all the results obtained is given in table 1. Column 1 of the table gives the date on which the observations were made, and column 2 gives the number of counts necessary to flash the neon tube of the integrating circuit. Column 3 shows the number of counts produced in each set of equipment by 1 mg of radium placed 1 m from the counters. Although the counters were made as nearly alike as was reasonably possible, there are evidently real differences in the sensitivities of the counters. These differences are made apparent by comparing the standard deviation of the values of column 3 from their mean with the deviation one would expect if an equivalent number of observations were taken with a single counter under constant conditions. The expected standard deviation of a single observation of N events, distributed at random (that is, according to

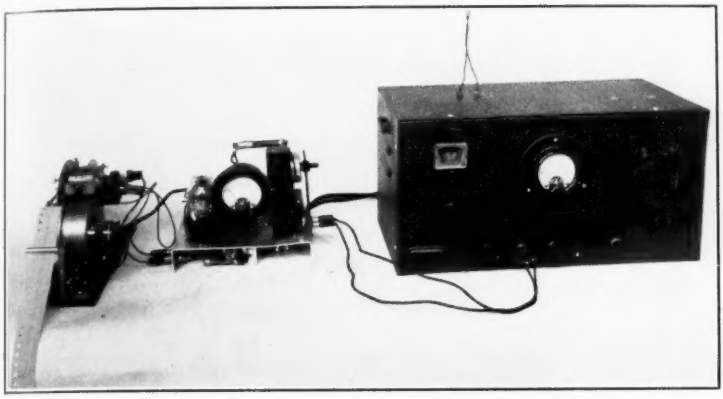

FIGURE 8.—*Complete receiving equipment.*

The superregenerative receiver is on the right. The detector circuit operated by the output of the receiver is in the center. The tape recorder is on the left.

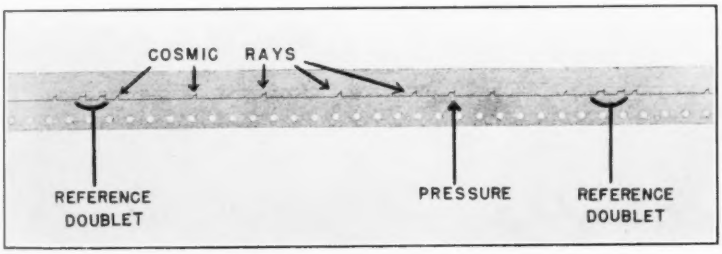

FIGURE 9.—*Sample of actual record on waxed tape.*

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Poisson's law), is $N^{1/2}$. The total number of events or counts involved is $3{,}320 \times 15 \times 5 = 249{,}000$ (since each of the 15 values in the table represents the average over 5 minutes). The expected standard deviation of this value would be 498 for the 75-minute period, which would give an expected, standard deviation of 7, in the average counting rate, per minute. This expected value of 7 for a single counter is so much less than the actual standard deviation, 110, of the 15 counters, from their mean that there is little doubt that real differences exist in the sensitivities of the counters.

TABLE 1.—*Summary and analysis of observations*

Date (1938)	Average counts per signal	Standardization, counts per minute for 1 mg of Ra at 1 m	Maximum observed counting rate in upper atmosphere, counts per minute	Pressure at altitude of maximum counting rate	Ratio of maximum counting rate to standard counting rate	Ratio of counting rate at 200 mb to maximum counting rate	Ratio of counting rate at 500 mb to maximum counting rate	Blank counting rate per minute	Pressure at maximum altitude attained	Ratio of counting rate at maximum altitude to maximum counting rate
1	2	3	4	5	6	7	8	9	10	11
				mb					*mb*	
7/13	40.6	3,830	2,110	60	0.55	0.50	.....	.....	15	0.79
7/15	52.0	3,540	2,340	60	.67	.62	.....	.....	15	.91
7/22	47.6	3,240	2,620	60	.81	.58	.....	.....	28	.86
7/25	40.1	2,690	4,160	55	.62	.62	.....	.....	15	.76
7/28	102.5	2,970	1,540	60	.62	.60	.....	.....	[a] 12	.73
9/27	49.6	3,120	1,850	50	.59	.59	0.12	25	[a] 8	[a] .88
9/29	72.8	3,280	1,610	60	.49	.59	.14	36	20	.89
10/9	49.1	3,280	1,590	55	.48	.63	.07	34	[a] 12	[a] .88
10/11	65.8	3,750	1,970	60	.53	.67	.07	130	[a] 8	[a] .75
10/12	63.6	3,490	1,930	60	.55	.57	.07	32	[a] 10	[a] .87
10/13	69.9	3,980	1,660	60	.42	.67	.04	140	[a] 10	[a] .77
10/17	46.0	3,580	1,670	55	.47	.68	.08	90	[a] 10	[a] .80
10/25	63.0	3,090	1,730	50	.56	.62	.04	50	[a] 10	[a] .84
10/26	59.0	2,600	1,440	60	.56	.57	.10	59	[a] 10	[a] .82
11/16	61.0	3,660	1,650	65	.45	.62	.11	91	35	.93
Average	.....	3,320	1,810	58	[b] 0.55	0.61	0.084	.....	[a] 10	[a] 0.82
Standard deviation	.....	110	85	1	[b] 0.023	0.012	0.033	.....	.....	0.02

[a] Only these values averaged.
[b] Omitting July 22, 1938, average=0.53; standard deviation=0.016.

In column 4 of table 1 is listed, for each sounding, the maximum observed counting rate obtained during the flight; and column 5 gives the average pressure, in millibars, observed at the time of maximum counting. Although the pressure could be read to the nearest 2 millibars, the pressure at the maximum counting rate could be estimated to only the nearest 5 millibars. The average value of the pressure at which the counting rate is a maximum is 58 millibars, which corresponds to an altitude of 69,500 ft. The percentage standard deviation of the values of column 4 from their average, 4.7 percent, is only slightly greater than for column 2, 3.3 percent. An increase of about this amount is not unexpected because of the increased uncertainties of measurement. It was hoped that by taking account of the differences in sensitivities of the various counters the deviations could be reduced. Accordingly, in column 6 are listed

the ratios of the maximum counting rates of column 4 to the standard counting rates of column 3. These ratios, with one exception, are noticeably more self-consistent than the maximum counting rates. Omitting the data of July 22, the percentage standard deviation of the ratios from their mean is less than 2 percent; including it the deviation is 4 percent. This one ratio is sufficiently out of line with the other 14 values to justify one of two conclusions, (1) something was wrong with the equipment, or (2) the cosmic-ray intensity at 60 millibars was abnormally high on July 22, 1938. There was no obvious inadequacy in the performance of the equipment, as is evident by referring back to figure 10, which is the record of the flight for this date. On the other hand, an examination of records from other laboratories has shown no departures from normal on this date of either the cosmic-ray intensity at sea level, or magnetic or solar phenomena. Accordingly, there is no reason to choose one conclusion over the other.

In column 7 are shown the ratios of the maximum counting rates to the counting at 200 millibars. The consistency of the data of this column merely indicates that the curves for all flights, such as shown in figure 8, had the same general shape. The agreement would probably be still better were it not for the facts (1), that counting rate was changing rapidly with time, and (2), that the counting time was extremely short. The data for 500 millibars, which are shown in column 8, are very erratic for not only are the counting rates varying and the counting period short, but the counting rate is low and appreciably influenced by the blank counting rate.

The blank counting rate represents the spontaneous discharge rate of the counter in the absence of radiation. It was determined by observing the counting rate in an iron shield of sufficient thickness to absorb gamma radiation and then subtracting from this value the number of counts expected from cosmic rays. Kolhörster and Janossy [9] have estimated the cosmic-ray intensity at sea level as 0.6 particle per cm^2 per minute, which value gives 19 counts per minute for our counters. This counting rate was then subtracted from the counting rates observed in the iron shield, and the remainder corresponds to the blank counting rates listed in column 9 of the table 1.

The minimum pressure attained in each flight is listed in column 10, and the ratio of counting rate at this pressure to the maximum counting rate is given in column 11. The minimum pressures recorded in nine of these flights differ by no more than the precision of reading, so the counting rates at minimum pressure have been averaged for these flights. This gives a counting rate at 10 millibars equal to 0.82 percent of the maximum counting rate. The very low counting rate obtained in one high-altitude flight in our earlier work has not been repeated, so it is probable that the earlier data were erratic.

Inasmuch as the barometric capsules showed uncertainties as large as ± 2 millibars during calibration, the error at 10 millibars may be as large as ± 20 percent. The altitude corresponding to 10 millibars is approximately 101,000 ft, and the uncertainty in reading the barograph means that the actual altitude attained may differ from this figure by approximately $\pm 5{,}000$ ft.

An estimate of the ratio of the maximum cosmic-ray intensity to sea-level intensity can be obtained by making use of the data of

[9] Z. Physik **93**, 119 (1935).

Kolhörster and Janossy referred to above. These data gave a sea-level counting rate of 19 for our counters, which is one ninety-fifth of the average value of the maximum counting rate, 1,810 per minute. This ratio is not directly comparable to similar ratios obtained by ionization chambers because of the directional effect of the counters. At sea level, where the cosmic rays are predominantly vertical, the effective area of the counters is essentially the product of the length and diameter. At high altitudes, however, observations such as those made in the stratosphere balloon *Explorer II* [10] have shown the vertical and horizontal components to be nearly equal. With such a directional distribution the average effective area of the counters used in our work has been found by integration to be very nearly 50 percent of the effective area to vertical radiation. This means that the ratio of our maximum count to ground count—that is, 95—should be approximately one-half the same ratio obtained by ionization-chamber measurements.

In the standardization of the counters with gamma radiation the radium was placed in the plane of the axis of the counters and at right angles to their axis, so that the effective area exposed to the gamma radiation was approximately one-half the effective area for vertical radiation or very nearly the same as the effective area to nondirectional radiation. Because of this it is believed that a reliable estimate of the maximum cosmic-ray intensity has been obtained by use of the standardized counters. This gives, as previously shown, a maximum counting rate equivalent to 0.54 mg of radium at a distance of 1 m.

WASHINGTON, August 8, 1939.

[10] W. F. G. Swann, C. L. Locher, and W. E. Danforth, Phys. Rev. **51**, 389 (1937).

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U. S. Department of Commerce National Bureau of Standards

RESEARCH PAPER RP1255

Part of Journal of Research of the National Bureau of Standards, Volume 23, November 1939

PHOTOCHEMICAL DECOMPOSITION OF THE CYSTINE IN WOOL

By Henry A. Rutherford and Milton Harris [1]

ABSTRACT

The work reported in the present paper is a continuation of earlier work on the photochemical decomposition of wool. The data confirm the hypothesis that the primary action of the radiation, which results in a change in the state of the sulfur and a corresponding lowering of the cystine content, is independent of the moisture in the system. The results indicate, however, that moisture plays an important role in the secondary process, namely that involving the evolution of hydrogen sulfide from the irradiated fibers. It is also shown that decomposition of the cystine resulting from thermal reactions under the conditions of these experiments is negligible.

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I. INTRODUCTION

In two earlier papers on the effect of light on wool [1, 2],[2] it was shown that one of the chief points of attack is the disulfide group of the amino acid, cystine. The data suggested that the primary action of the radiation is an activation of the disulfide group, resulting in a change in state of the sulfur and a corresponding lowering of the cystine content. A secondary process, involving the evolution of hydrogen sulfide from the irradiated wool, then occurs. The rate of the primary reaction, that is, the decomposition of the cystine, appeared to be independent of the amount of water vapor in the system surrounding the fibers during irradiation. It was believed, however, that water might play an important part in the secondary reactions. The work reported in the present paper is a continuation of the earlier investigation.

[1] Research Associates at the National Bureau of Standards, representing the Textile Foundation, Inc.
[2] Figures in brackets indicate the literature references at the end of this paper.

II. EXPERIMENTAL PROCEDURE

The apparatus, with the exception of a few minor changes described below, and technique of operation were the same as described in detail in an earlier paper [2]. The wool samples were irradiated in specially constructed tubes, in atmospheres of moist and dry nitrogen. The nitrogen, which was used as a carrier gas to remove the hydrogen sulfide formed during the irradiation, was first freed from oxygen by passing it through two towers of alkaline pyrogallol. The emerging gas was divided into two portions, one of which was moistened by passing it through distilled water, while the remainder was dried over anhydrous magnesium perchlorate and phosphorus pentoxide. From the drying and moistening towers, the gas passed through the exposure tubes and finally into an alkaline solution of bromine for absorption of hydrogen sulfide. The smooth elliptical aluminum reflector, used around the lamp and exposure tubes in the original apparatus, was replaced by a corrugated reflector in order to insure a more nearly uniform irradiation of the fibers. A stopcock was inserted between the tubes emerging from the drying and moistening towers so that both exposure tubes could be supplied with moist or with dry nitrogen at the same time.

Single-ply wool yarn of very low twist was used for the exposures. The yarn was prepared from raw wool which had been extracted with Stoddard solvent and washed with water. It was further purified by extraction in a Soxhlet apparatus with alcohol and ether, for 6 hours each, and finally washed with water at 50° C.

1. EFFECT OF LIGHT ON THE CYSTINE IN WOOL

Twenty 100-mg portions of the yarn were wound in adjoining layers on each of the two hexagonal glass frames. The wool on one frame was dried in an oven at 105° C for 3 hours, then rapidly transferred to the exposure tube, where it was further dried in a stream of dry nitrogen at 90° to 100° C for 24 hours. The exposures were then started, dry nitrogen being passed over the dried sample and moist nitrogen over the other. During the exposures, the frames were rotated as described earlier [2]. After different time intervals, two of the 100-mg skeins, one from the top and one from the bottom of each frame, were removed from each of the exposure tubes and analyzed for cystine by the Sullivan method [3], using the hydrochloric-formic acid mixture for hydrolysis, as described by Miller and du Vigneaud [4]. The cystine contents of the samples after the exposures are shown in table 1.

TABLE 1.—*Cystine content of wool after irradiation in atmospheres of moist and of dry nitrogen*

Duration of exposure	Moist nitrogen		Dry nitrogen	
	Cystine	Sulfur	Cystine	Sulfur
Hour	*Percent*	*Percent*	*Percent*	*Percent*
0	13.2	3.57	13.2	3.57
98	10.1		10.1	
196	8.2		9.2	
299	7.0		8.0	
394	5.7		6.8	
493	5.2	2.99	6.3	3.35

2. EFFECT OF TEMPERATURE

The temperature in the exposure tubes, as measured by a mercury thermometer (the bulb of which was covered with a thin layer of wool yarn), varied between 65° and 70° C. Since the temperature coefficient of most photochemical reactions is known to be small compared with that of most thermal reactions, it is hardly likely that the rate of decomposition of the cystine was appreciably affected by this small variation in temperature.

In order to determine whether some of the cystine was destroyed by thermal reactions alone during the long exposures, samples of wool were heated in the dark in atmospheres of moist and dry nitrogen at 60° and 105° C. An experiment was run at the higher temperature, since it was conceivable that the temperature on the surface of the fibers might be somewhat higher than that indicated by the thermometer. The wool yarn was placed in glass tubes in an oven; and nitrogen, dried or moistened by passing through towers of phosphorus pentoxide or water, was passed over the samples at the rate of about 3 liters per hour. The nitrogen was preheated to the desired temperature, after leaving the towers, by allowing it to pass through about 12 ft. of copper coil in the oven. After different time intervals, duplicate samples were removed for sulfur and cystine analyses, the results of which are shown in table 2.

TABLE 2.—*Effect on the sulfur and cystine in wool of prolonged heating in the dark*

Temperature	Duration of heating	Total sulfur		Cystine	
		moist	dry	moist	dry
°C	hr	%	%	%	%
	[a] 0	3.55	3.55	12.7	12.7
60	210	3.57	3.60	13.3	13.3
60	500	3.53	3.56	13.4	13.4
105	192	3.55	3.56	12.5	12.4
105	408	3.55	3.52	11.7	12.3
105	690	3.50	3.55	12.2	11.7

[a] Untreated wool.

3. NATURE OF THE PRIMARY AND SECONDARY REACTIONS

Samples of the wool yarn were wound on the glass frames and irradiated in atmospheres of moist and dry nitrogen for 200 hours. The lamp was then turned off, the temperature maintained at 70°C, and moist nitrogen passed over both samples for about 300 hours. Finally, the lamp was again turned on and both samples were irradiated in atmospheres of moist nitrogen for an additional 300 hours. The rates at which sulfur, in the form of hydrogen sulfide, is evolved from the wool are shown in figure 1.

III. DISCUSSION

The data recorded in table 1, showing the relative rates of decomposition of cystine during irradiation in atmospheres of moist and of dry nitrogen, confirm the earlier observation that the primary action of the radiation, which results in the decomposition of the cystine, is independent of the moisture in the system. The cystine contents

of the samples irradiated in atmospheres of moist nitrogen are slightly lower; but in view of experimental difficulties, such as keeping the intensity of the light on both samples equal [3] and constant, these small differences cannot be considered as significant at this time. Furthermore, the differences are extremely small when compared with the difference between the rates of evolution of hydrogen sulfide from wool irradiated in atmospheres of moist and of dry nitrogen.

The decomposition of the cystine under the conditions of these experiments appears to result from photochemical rather than thermal reactions. As was pointed out previously, the temperature in the

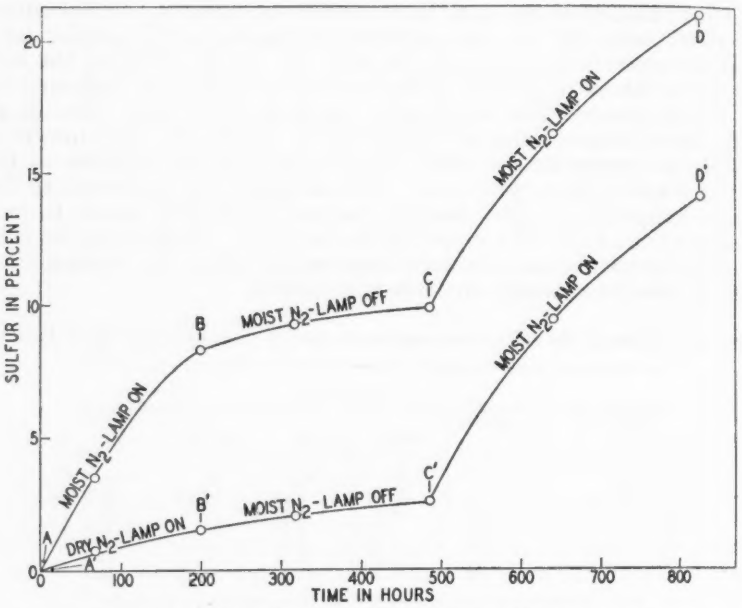

FIGURE 1.—*Rate of evolution of hydrogen sulfide from wool.*

The measurements were made (1) during irradiation in atmospheres of moist and dry nitrogen, (2) in the dark in atmospheres of moist nitrogen, the temperature being maintained at 70°C, and (3) during irradiation in atmospheres of only moist nitrogen.
The ordinate values are in terms of the percentage of the total sulfur which was evolved as hydrogen sulfide during the treatment.

exposure tube varied between 65° and 70° C. However, the estimation of the exact temperature on the surface of the fiber would be very difficult, and for that reason an experiment in the absence of light was also run at 105° C. The data in table 2 show that no significant changes in the sulfur or cystine contents of the wool occurred during the prolonged heating at 60° and 105° C.

Although the presence of moisture in the atmosphere surrounding the fibers during irradiation is not required for the primary reaction involving the decomposition of the cystine, the present work indicates that it does play an important role in the secondary process involving the evolution of the hydrogen sulfide from the irradiated fibers.

[3] When samples were irradiated under the same conditions in both exposure tubes, hydrogen sulfide was evolved at the same rate. This was the basis for assuming equal intensity during subsequent experiments.

Furthermore, it should be emphasized that the primary reaction is much more rapid than the secondary reaction and that the rate of evolution of hydrogen sulfide from the wool is determined by the rate of the secondary process. In addition, it appears that the secondary process is essentially a photochemical one, as indicated by the *BCD* and *B′C′D′* portions of the curves in figure 1. That a small amount of hydrogen sulfide is probably split from irradiated wool by thermal processes is indicated by the slopes of *BC* and *B′C′*, but a comparison with those of the *CD* and *C′D′* portions shows that the amount is extremely small.

The authors can, at present, offer no complete explanation of the photochemical processes involved in the decomposition of the cystine in wool. The results to date suggest that the initial state of the photochemical decomposition is an absorption of the radiant energy resulting in the dissociation of the R–S–S–R group, probably into $R\text{–}S^+$ and $R\text{–}S^-$ ions. This reaction does not appear to be catalyzed by moisture. A cleavage of this type for the photochemical decomposition of cystine itself has been postulated by Anslow and Foster [5], who showed that when radiant energy is absorbed by this substance, the linkage between the halves of the molecule is weakened, which results in the disruption of the molecule. A possible secondary reaction would then be the reaction of the products of the primary process with water to form RSH and RSOH compounds. The latter are unstable and generally decompose with the liberation of hydrogen sulfide.

IV. REFERENCES

[1] A. L. Smith and M. Harris, J. Research NBS **17,** 97 (1936) RP904.
[2] M. Harris and A. L. Smith, J. Research NBS **20,** 563 (1938) RP1091.
[3] M. X. Sullivan, Public Health Reports No. **86** (1930).
[4] G. Miller and V. du Vigneaud, J. Biol. Chem. **118,** 101 (1937).
[5] G. A. Anslow and M. L. Foster, J. Biol. Chem. **97,** 37 (1932).

Washington, August 31, 1939.

U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

RESEARCH PAPER RP1256

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TENSILE PROPERTIES OF RUBBER COMPOUNDS AT HIGH RATES OF STRETCH

By Frank L. Roth and William L. Holt

ABSTRACT

With the aim of determining whether the tensile properties of a rubber compound measured at high stretching rates offer any better indication of resistance to abrasive wear than the properties measured at the usual testing rates, a special apparatus was developed for stretching ring specimens to rupture by means of a falling weight. Tensile properties of the ring specimens were determined from a study of the position of the weight as a function of time. The position of the weight was recorded at equal time intervals on a paper tape, one end of which was fixed to the weight. The tape was pulled through a spark gap as the weight fell. The timing of the sparks was controlled by a tuning fork. Specimens were stretched to the point of rupture in about one-half to three quarters of a second. The work of extension was determined as a function of the elongation of the specimen from the instantaneous positions and corresponding speeds of the weight. Stress-strain relationships were then obtained from the work of extension.

Tests were made on a range of cures of each of four rubber compounds. One of these was a pure gum compound, and each of the other three contained 25 volumes of a filler per 100 volumes of crude rubber. The three fillers used were channel black, soft black, and clay.

A comparison of these data with similar data for a rate of stretch which lies in the range of speeds common to the usual routine tests fails to show that the tensile properties measured at the high stretching rates offer any better indication of resistance to abrasive wear than the properties measured at the usual testing rates.

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I. INTRODUCTION

It has been suggested by several authors that some relationship may exist between the stress-strain properties of a rubber compound and its resistance to abrasive wear. Wiegand [1] pointed out in 1920 that since

[1] W. B. Wiegand, *Some aspects of the rubber stress-strain curve*, Can. Chem. J. 4, 160 (1920).

the wear of a tire tread consists in the gouging or tearing out of small masses of rubber, the resistance to abrasive wear might depend on the amount of work performed in stretching the rubber to rupture. A chart accompanying this suggestion showed that for a compound containing 25 volumes of channel black per 100 volumes of rubber this work was greater than for compounds containing other fillers or other proportions of channel black. It is well known that compounds containing from 25 to 35 volumes of channel black have the highest resistance to abrasive wear. Grieder [2] found no exact proportionality between the work of extension and abrasive resistance obtained in the laboratory, but compounds which showed high resistance to abrasion also showed relatively large work of extension. In a more recent investigation on the reinforcement of rubber with carbon black, Wiegand [3] states that the resistance of a rubber compound to abrasive wear is undoubtedly influenced not only by the work of extension to rupture but also by the work of extension to intermediate elongations.

Principally because of experimental difficulties in obtaining simultaneous values of extension and force at high rates of stretch, most of the previous investigations have been limited to relatively slow rates. However, it has long been known that the tensile properties of a vulcanized rubber compound depend on the rate at which the specimens are stretched. In 1903 Bouasse and Carrière [4] noted that for a rubber-sulfur compound the extension of a specimen for a given load was greater when the load was applied slowly than when it was applied quickly. The extensions were carried out to about 600 percent of the unstretched length of the specimens at rates ranging from 0.5 to 7 percent per second.[5] Somerville, Ball and Edland [6] noted for a more modern pure-gum compound a similar effect of stretching rates from 0.3 to 3 percent per second and for extensions to 350 percent.

Holt,[7] using an autographic machine, stretched a tire-tread compound to an elongation of 450 percent at average rates ranging from 10 to 1,500 percent per second. His results also show that for any given strain the stress increases with increasing rate of stretch. Similar results were obtained by Dart and Guth,[8] who used a method involving photographic recording and employed rates ranging from 2 to 300 percent per second.

The effects of average rates of extension from about 2 to 20 percent per second on the tensile strengths and ultimate elongations for four compounds were reported by the National Bureau of Standards[9] in 1915. The study showed slightly higher tensile strengths and ultimate elongations for the higher rates of stretch.

The above-mentioned investigators have found small increases in the stress for a given strain, but no one of them has reported any marked advantage in making tests at the higher rates of stretch.

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[2] H. W. Grieder, *The resilient energy and abrasion resistance of vulcanized rubber*, Ind. Eng. Chem. **15**, 504 (1923).

[3] W. B. Wiegand, *The carbon reinforcement of rubber*, Can. Chem. and Met. **21**, 35 (1937).

[4] H. Bouasse and Z. Carrière, *Sur les courbes de traction du caoutchouc vulcanisé*, Ann. Faculté Sciences Toulouse, **5** (2), 257 (1903).

[5] The rate of stretch for the usual dumbbell specimens ranges from 5 to 25 percent per second for a jaw separation of 20 inches per minute.

[6] A. A. Somerville, J. M. Ball, and L. A. Edland, *Autographic stress-strain curves of rubber at low elongations*, Ind. Eng. Chem., Anal. Ed. **2**, 289 (1930); Reprinted in Rubber Chem. Tech. **3**, 689 (1930).

[7] W. L. Holt, *Behavior of rubber under repeated stresses*, Ind. Eng. Chem. **24**, 1471 (1931); Reprinted in Rubber Chem. Tech. **5**, 79 (1932).

[8] F. E. Dart and E. Guth, *The dependency of the stress-strain relationship for rubber upon the rates of stretching*, Phys. Rev. **55**, 1141 (A) (1939).

[9] Cir. BS C38 [ed. 3], 60 (1915).

Beadle and Stevens [10] suggested the use of the impact pendulum for stretching rubber test specimens in order that the tests be made to approach more nearly the sudden stresses experienced by a tire on the road. Later studies by investigators [11] [12] using the impact pendulum to determine the work done in stretching rubber to rupture report that high rates of stretch are advantageous in determining the best cure. Stress-strain curves obtained for elongations up to 350 percent at a rate of about 8,000 percent per second by means of a modified impact pendulum [13] show stresses which are relatively large compared with those obtained by the usual methods of testing.

In a recent investigation of isothermal and adiabatic stress-strain curves for pure-gum compounds, Hauk and Neumann [14] found that for compounds containing 1 and 2 percent of sulfur "the curves first separate, then at higher elongations approach each other, and finally intersect." In this work the isothermal curves were determined by loading similar strip-specimens with various weights and observing the change in distance between gage marks. The adiabatic curves were determined by applying the stretching force through a calibrated spring and obtaining a cinematographic record of the relative positions of gage marks on both the specimen and the spring. The stretching process for the adiabatic curves was accomplished in less than a second. The same authors,[15] working with elongations up to about 450 percent, found in another investigation that for various rates of stretch ranging from 50 to 700 percent per second the stress increased with increasing rates.

The purpose of the present investigation was to determine whether the tensile properties measured at high stretching rates offer any better indication of resistance to abrasive wear than the properties measured at the usual testing rates. In this study stress-strain relationships were determined for different types of rubber compounds when stretched rapidly to rupture. The specimens were stretched to rupture in less than a second, so that the tests were nearly adiabatic. The cross sections of the specimens were small compared to their lengths so that the stresses might approach simple tensions in character. In order to make the results for high rates of stretch comparable to those for low rates, similar specimens were used throughout the study.

II. RUBBER COMPOUNDS INVESTIGATED

Tests were made on a series of specimens of each of four rubber compounds, the compositions and cures of which are shown in table 1. The first compound is referred to as pure gum, that is, it contains no filler. The compounds containing fillers, referred to as loaded compounds and designated by their respective fillers, have a base stock which is as nearly like the pure-gum compound as is practicable.

[10] C. Beadle and H. P. Stevens, *Impact tensile tests on rubber and a comparison with tensile and hysteresis tests*, Proc. Int. Rubber Cong. (London) **4**, 344 (1911).
[11] A. van Rossem and H. B. Beaverdam, *Tensile tests of vulcanized rubber at high speed*, Rev. gén. caoutchouc **7**, 27 (1930); Reprinted in Rubber Chem. Tech. **4**, 147 (1931).
[12] G. W. Albertoni, *Impact machine for rubber testing*, Ind. Eng. Chem. **9**, 30 (1937); Reprinted in Rubber Chem. Tech. **10**, 317 (1937).
[13] G. W. Albertoni, *Impact machine for rubber testing*, Ind. Eng. Chem. **9**, 30 (1937); Reprinted in Rubber Chem. Tech. **10**, 317 (1937).
[14] V. Hauk and W. Neumann, *Isothermal and adiabatic stress-strain curves of vulcanized rubber*, Monatsh. **72**, 22 (1938); Translated in Rubber Chem. Tech. **12**, 64 (1939).
[15] V. Hauk and W. Neumann, *A time effect in the rapid elongation of rubber*, Naturwissenschaften **26**, 365 and 461 (1938); Translated in Rubber Chem. Tech. **12**, 518 (1939).

Each compound contains 25 volumes of filler per 100 volumes of crude rubber.

TABLE 1.—*Composition and cure of rubber compounds*

Rubber compounds	Pure-gum	Channel-black	Soft-black	Clay
Ingredients	Parts by weight			
Rubber (smoked sheets)	100	100	100	100
Sulfur	3	3	3	3
Zinc oxide	5	5	5	5
Stearic acid	1	2	2	2
Benzothiazyl disulfide (Altax)	1.0	1.0	1.0	1.0
Tetramethylthiuram disulfide (Tuads)	----	0.1	0.1	0.1
Phenyl-beta-naphthylamine	1.5	1.5	1.5	1.5
Channel-black (Micronex)	----	49.0	----	----
Soft-carbon (P–33)	----	----	49.0	----
Clay (Dixie)	----	----	----	70.7
Curing temperature (° C)	142	127	127	127
Curing times (min)	20	20	10	20
	25	25	[a] 15	25
	[a] 30	[a] 35	20	[a] 35
	40	50	25	50
	60	75	35	75
	75	----	----	----

[a] Cures selected as optimum for comparisons in the present investigation.

In the selection of the compounds to be studied, the aim was to pick a few simple compounds containing commonly used fillers and differing considerably in tensile and abrasive properties. The addition of channel black to a base stock produces a compound which possesses the greatest resistance to abrasive wear, and which is universally used for tire treads. The compound is stiff and has markedly higher tensile strength than the base stock. When clay is added to the base stock, a stiff compound with relatively low tensile strength and ultimate elongation results. Soft black, however, has little stiffening action, and a soft-black compound has a relatively high ultimate elongation. Such a compound has a tensile strength less than that of the channel-black compound but more than that of the clay compound.

III. APPARATUS AND PROCEDURE

The tensile properties for high rates of stretch were determined by means of a special apparatus in which ring specimens were stretched by means of a falling weight. Slow stretching rates were obtained by use of a modified form of this apparatus and by a Scott tensile machine.

1. RAPID STRETCHING

(a) DESCRIPTION OF APPARATUS

A schematic diagram of the apparatus employed in obtaining stress-strain relationships for high rates of elongation is shown in figure 1. Ring specimens are stretched to rupture by a falling weight, and their stress-strain relationships are determined from a study of the position of the weight as a function of time. The position of the weight is recorded at equal time intervals on a paper tape, one end of which is fixed to the weight. The tape is pulled through a spark

gap as the weight falls. The timing of the sparks is controlled by a tuning fork which operates the breaker points in the primary circuit of an induction coil. The frequency of the tuning fork is 55.6 vibrations per second, and the time required to stretch a specimen to the

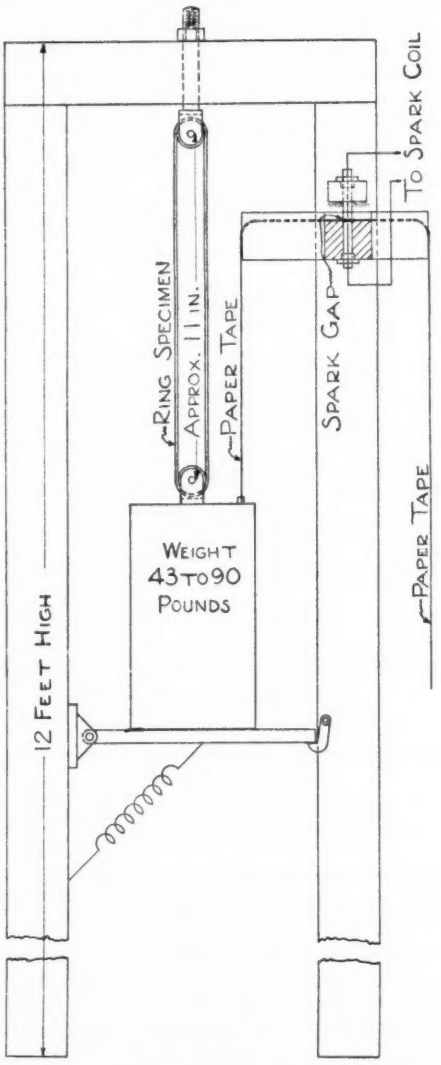

FIGURE 1.—*Schematic diagram of the apparatus for determining the tensile properties of rubber by the use of a falling weight.*

point of rupture ranges from about one-half to three-quarters of a second. The time involved in stretching the specimen is short enough so that the test may be considered nearly adiabatic. The rate of stretch is not constant, but the average is in the order of 1,000 percent per second. The maximum speeds range from 1,600 to 1,950 percent per second for the pure-gum compounds and from 950 to

1,500 percent per second for the loaded compounds. The actual rates of stretch for a pure-gum and a loaded compound are shown in figure 2. The fact that the speed for the channel-black specimen selected for this figure does not start from zero indicates that the weight fell some distance before beginning to stretch the specimen. In the case of the pure-gum specimen selected, this distance was zero, and therefore the speed starts from zero. The channel-black and pure-gum specimens selected here were stretched by weights of 90.2 and 42.93 pounds, respectively. Variations in the rate of stretch of the specimen, brought about by changes in the weight or in the distance the weight falls before beginning to stretch the specimen, have no observable effect on the data obtained.

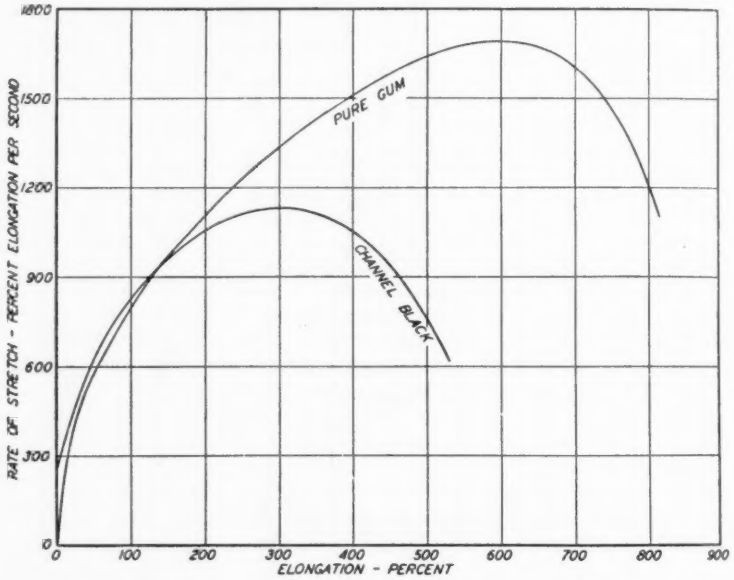

FIGURE 2.—*Rates of stretch for typical specimens of channel-black and pure-gum compounds when stretched by the falling weight.*

The molded-ring specimens are of rectangular cross section with inside and outside diameters of about 7 and 7¼ inches, respectively, and a width of 0.2 inch. The difference between the inside and outside diameters was made relatively small in order to minimize the difference between the elongations of the inner and outer surfaces of the rings during the stretching process. The specimens are supported on two steel spools, ⅞ inch in diameter, one of which is fixed in position and the other is part of the falling weight. The spools are free to rotate on their axes, and the areas of contact between the ring and the spools are lubricated with castor oil. Except for the highly cured pure-gum compounds, few specimens rupture in or very near the region of contact of the rings with the spools.

The weight is made in several sections so that it can be varied between 43 and 90 pounds by steps of approximately 12 pounds. The upper, or fixed, spool may be raised or lowered to allow the

weight to fall any desired distance (usually from 0 to 2 inches) before beginning to stretch the specimen.

The spark electrodes consist of tungsten wires inside glass capillary tubes. The bore of these tubes is about 0.020 inch, and the electrode ends of the wires reach to within about 0.010 inch of the ends of the tubes. The recording tape slides between the tubes, and the tubes are kept in contact with it by the weight of the upper electrode assembly. Strips of adding-machine paper, 1¼ inches wide and 10 feet long, are used for the recording tape. A flat spring keeps the tape taut as the weight falls. The friction of the tape produces no appreciable effect on the motion of the weight since, if the weight is allowed to fall without a specimen in place, its acceleration is found to be equal, within an experimental error of about 1 part in 300, to the accepted value of g, the acceleration of a freely falling body.

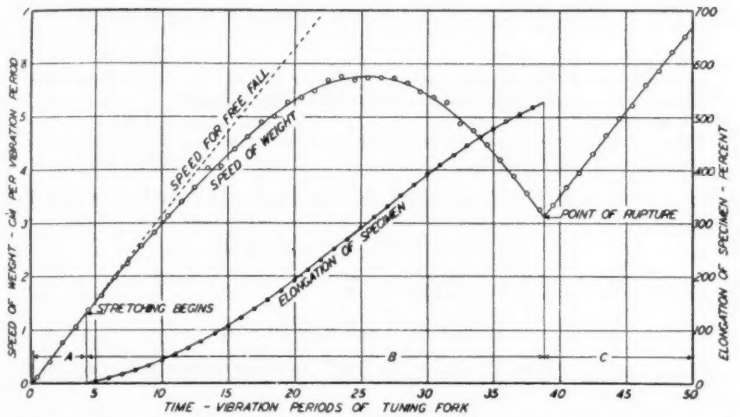

FIGURE 3.—*Speed-time and elongation-time curves for a specimen of channel black compound stretched by the falling weight.*

The positions of the spark holes in the tape are measured by means of a low-power microscope and a millimeter scale. The distance between any two consecutive holes, after the starting point, is numerically equal to the average speed of the weight during the corresponding time interval and is equal, to a first-order approximation, to the instantaneous speed at a time midway between the beginning and end of the interval. Figure 3 shows a typical speed-time graph plotted by this method. Region A corresponds to the free fall of the weight before it begins to stretch the specimen. The distance of free fall was found by direct measurement before the weight was released, and in this case was 1.1 inches (2.8 cm). Region B represents the fall of the weight under the action of the upward pull of the specimen. Region C represents the free fall of the weight following the rupture of the specimen.

The tensile properties of the specimen may be determined in either of two ways: (1) The tension in the specimen may be determined at any point during its stretch from the speed-time curve; or (2) the work done by the weight on the specimen may be determined and

plotted as a function of its elongation, and the tensile properties may be determined from the resulting graph. In the first method the tension, especially for low elongations, must be obtained from the difference of two relatively large quantities. Consequently, this method was abandoned in favor of the less direct method, that of considering the work done by the weight on the specimen.

Since data obtained by allowing the weight to fall without a specimen in place show that frictional forces due to the tape are negligible, the work done on the specimen is equal to the difference between the change in potential energy of the weight due to its change in position and the kinetic energy due to its speed. The curves in figure 3 are needed only to determine the speed of the weight as a function of the elongation of the specimen and to determine the ultimate elongation of the specimen.

(b) SAMPLE CALCULATION

A sample calculation for the determination of the work done in stretching the specimen represented by figure 3 is as follows:

Weight	90.2 lb
Frequency of the tuning fork	55.6 cycles/sec
Distance weight falls before beginning to stretch the specimen	1.1 in.
Half-circumference of the unstretched specimen	11.15 in.
Volume of the specimen	0.583 in.^{3}

For an elongation of 350 percent, for example, the distance the weight has fallen is

$$(11.15\times 3.5)+1.1=40.1 \text{ in.}$$

The change in its potential energy is

$$90.2\times 40.1=3{,}620 \text{ in.-lb.}$$

Its speed, taken from figure 3, is 5.67 cm per vibration period of the tuning fork, or

$$\frac{5.67\times 55.6}{2.54}=124 \text{ in./sec.}$$

The gravitational constant is $980/2.54=386$ in./sec^2.
The kinetic energy of the weight is given by

$$\frac{90.2\times (124)^2}{2\times 386}=1{,}800 \text{ in.-lb.}$$

The work done per cubic inch of the specimen in stretching 350 percent is

$$\frac{3{,}620-1{,}800}{0.583}=3{,}120 \text{ in.-lb./in}^3.$$

Similar calculations may be carried out for as many elongations as may be desired. Figure 4 shows the work done per unit volume of the sample plotted as a function of the elongation. Since for any given elongation the stress is equal to the ratio of the change in work done per unit volume to the corresponding change in strain, the slope at any point of the work curve, figure 4, is equal to the stress at the corresponding strain. Therefore the stress-strain curve for the speci-

men can easily be plotted either from an analytical treatment of the work data or by graphical means. Both methods were employed in the present investigation.

(c) PRECISION

The precision of the measurements for high rates of stretch is as good as can be obtained by the usual testing methods. Tables 2 and 3 show the work of extension at various elongations of pure-gum and

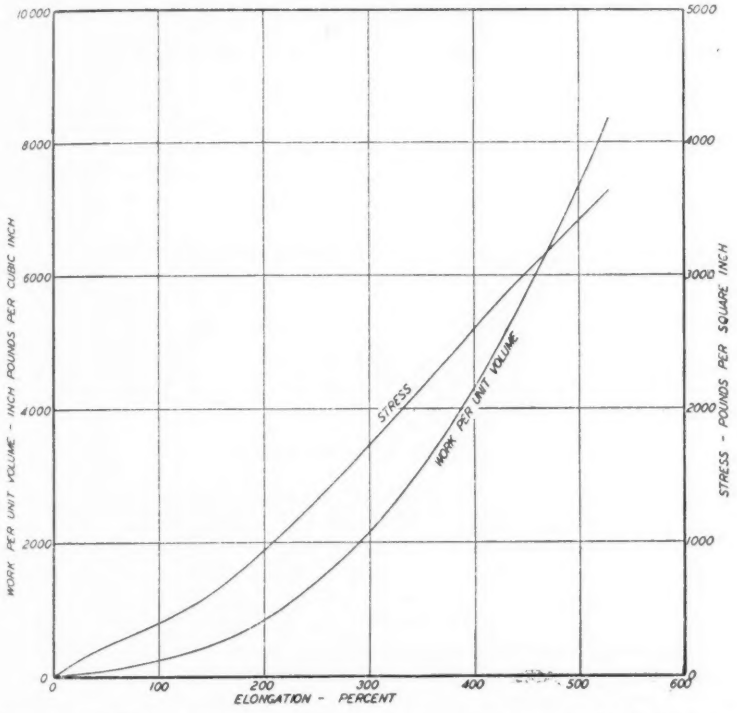

FIGURE 4.—*Work-elongation curve and the stress-strain curve obtained from it.*

The work-elongation curve is derived from figure 3.

channel-black compounds at optimum cure and are illustrative of the general precision obtained. The coefficient of variation used in these tables is defined as the ratio of the standard deviation to the mean, the standard deviation being the square root of the mean of the squares of the deviations of the individual observations from their mean. The coefficients of variation observed at rupture are in good agreement with the value 0.05 stated by Holt and McPherson [16] to be that commonly obtained by the usual testing methods.

[16] W. L. Holt and A. T. McPherson, *Toggle clamp for rubber tensile specimens*, J. Research NBS **22**, 543 (1939) RP1204.

TABLE 2.—*Work of extension for the pure-gum compound at optimum cure*

Elongation (percent)	50	150	250	350	450	550	650	750	Rupture
Ring No.	Work of extension, inch-pounds per cubic inch								
257	26	115	274	491	837	1,390	2,265	4,000	5,340
259 *									5,510
261	25	124	287	498	840	1,380	2,350	4,130	5,070
262	22	114	279	507	808	1,355	2,270	4,030	5,900
263 *									5,520
264	23	120	280	498	828	1,315	2,260	4,080	5,920
Average	24	118	280	498	828	1,360	2,285	4,060	5,543
Coefficient of variation	0.066	0.034	0.017	0.011	0.015	0.021	0.016	0.012	0.051

* Values were not calculated for work of extension at elongations below rupture.

TABLE 3.—*Work of extension for the channel-black compound at optimum cure*

Elongation (percent)	100	200	300	400	500	Rupture
Ring No.	Work of extension, inch-pounds per cubic inch					
87	216	840	2,140	4,290	7,310	8,330
88	214	859	2,194	4,360	7,456	9,235
89	215	833	2,130	4,270	7,360	9,170
Average	215	844	2,155	4,307	7,375	8,910
Coefficient of variation	0.004	0.013	0.013	0.009	0.008	0.046

2. SLOW STRETCHING

In order to eliminate the effect of variables other than the rate of stretch, identical specimens were made up for both rates. The stress-strain relationships for slow stretching were determined for all but the pure-gum compounds on a Scott tensile machine, using sections of the rings as strip specimens and toggle clamps [17] to hold the strips. With 2-inch gage marks and a jaw separation of 20 inches per minute, the speed of stretch is 8 to 10 percent per second. In order to determine whether the geometry of the specimen or the clamping arrangement affected the observed tensile properties, a number of rings of a carbon-black compound were stretched at about the same rate, 8 to 10 percent per second, over the lubricated spools used for rapid stretching. The resulting stress-strain curves, including the average ultimate elongations and tensile strengths were not appreciably different from those obtained with strip specimens of the same compound. Since strips from the pure-gum rings showed a decided tendency to break in or near the clamps, all the data for this compound were obtained by stretching complete rings over the lubricated spools.

[17] W. L. Holt and A. T. McPherson, *Toggle clamp for rubber tensile specimens*, J. Research NBS 22, 543 (1939) RP1204.

IV. EXPERIMENTAL RESULTS

1. STRESS-STRAIN RELATIONSHIPS

Stress-strain curves for the optimum cure of each compound are shown in figure 5. In each case the solid line refers to rapid stretching and the broken line refers to slow stretching. The data corresponding to the points shown are tabulated in table 4. It will be noted that

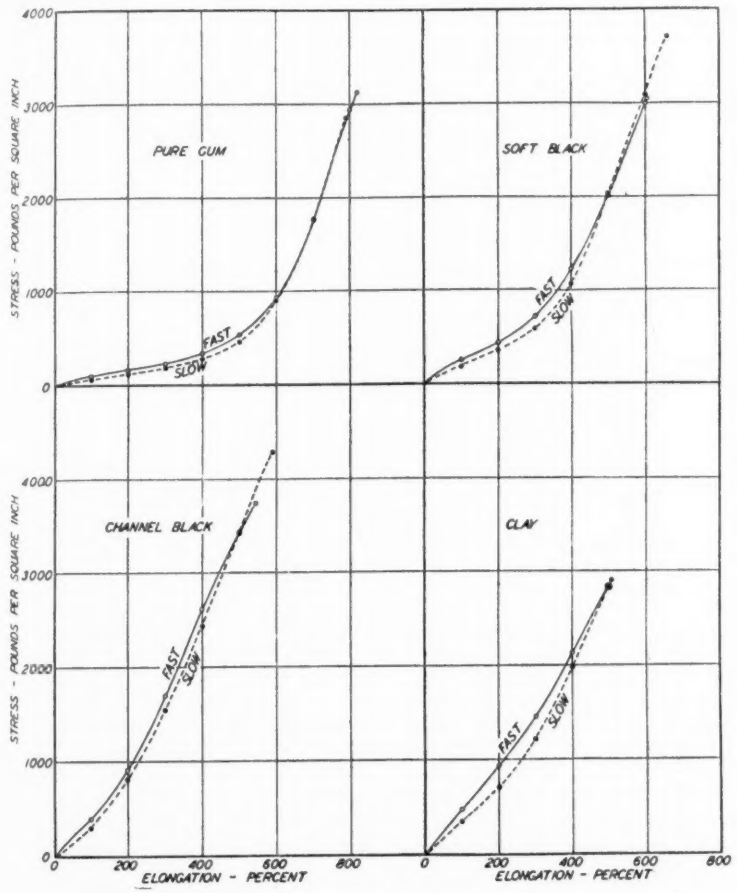

FIGURE 5.—*Comparative stress-strain curves for two rates of stretch.*
The curves are based on the optimum cures of the compounds described in table 1.

for elongations up to about 500 or 600 percent the stress at a given elongation is greater for rapid stretching. For the pure-gum compound the difference in the stresses for the two rates of stretch reaches a maximum of 75 pounds per square inch at 500-percent elongation. At elongations greater than 600 percent the difference is negligible. For the loaded compounds the difference is somewhat greater but vanishes at about 500-percent elongation and reverses its sign at higher elongations.

TABLE 4.—*Comparison of stresses of rubber compounds at optimum cure for the two rates of stretch*

Compound	Pure gum		Soft black		Channel black		Clay	
Rate of stretch	Fast	Slow	Fast	Slow	Fast	Slow	Fast	Slow
Elongation	Stress, pounds per square inch							
Percent								
100	92	56	250	180	390	295	470	345
200	161	107	435	355	910	810	940	705
300	220	173	705	580	1,700	1,540	1,460	1,215
400	329	270	1,225	1,070	2,610	2,430	2,130	1,980
500	530	455	2,010	2,025	3,430	3,415	2,840	2,840
600	912	895	2,990	3,085	------	------	------	------
700	1,770	1,760	------	------	------	------	------	------
Tensile strength	3,120	2,850	3,040	3,705	3,740	4,280	2,840	2,900

The fact that the curves for high rates of stretch approach or cross those for slow stretching is in accord with the observations of Hauk and Neumann.[18] This effect might be accounted for, at least in part, by the greater increase of temperature of the specimen when it is stretched more quickly. This increase of temperature at high stretching rates was greater for the loaded compounds than for the pure-gum. Specimens of the channel-black compound were found, about 15 seconds after rupture, to be at temperatures as much as 25° C higher than they were before stretching began. A study of the effect of the temperatures of several rubber compounds on their respective tensile stresses, reported by the National Bureau of Standards,[19] shows that for given elongations the tensile stresses of rubber compounds decrease with increasing temperatures. This effect is greater for loaded compounds than for pure gum.

Figure 6 presents the tensile characteristics of the compounds as a function of the curing time. Although the actual values of the stresses differ for the two rates of stretch, their general variations with changes in cure differ but little. In regard to the tensile strengths for the pure-gum compound, it should be mentioned that for cures greater than 30 minutes all the ruptures occurred at or near the supporting spools.

Except for the pure-gum compound and the channel-black compound of lowest cure, the tensile stresses and elongations at rupture are less for rapid than for slow stretching. The differences in the behavior of the loaded and pure-gum compounds at the two rates of stretch are probably due to differences in (1) the increase of the temperatures referred to above, (2) the behavior of the specimen at the region of contact with the supporting spools, and (3) the rate and type of fibering [20] of the different compounds. No attempt is made to explain the exact causes of the observed differences.

[18] V. Hauk and W. Neumann, *Isothermal and adiabatic stress-strain curves of vulcanized rubber*, Monatsh. **72**, 22 (1938); Translated in Rubber Chem. Tech. **12**, 64 (1939).

[19] R. F. Tener, S. S. Kingsbury, and W. L. Holt, *Tensile properties of soft rubber compounds at temperatures ranging from −70° to +147° C.*, Techn. Pap. BS **22**, 367 (1928) T364.

[20] Fibering or "crystallization" of rubber compounds is a well-known phenomenon of phase change which occurs in rubber when it is stretched. This phase change is evidenced by changes in X-ray diffraction patterns, specific volumes, and by specific heats. Fibering is influenced by differences in compounds, amount and rate of stretch of the specimens, and by temperature. In effect, the stress-strain curve for low elongations represents the tensile properties of an amorphous substance, whereas the same curve for higher elongations may represent the tensile properties of a type of crystalline substance. Thus, it is difficult to predict the exact causes of the observed differences in tensile strengths for the various compounds when they are stretched at different rates.

2. WORK OF EXTENSION

Table 5 shows the work of extension of the rubber compounds at optimum cure tabulated as a function of elongation. For the compounds investigated the ratio of the work of extension for slow stretch-

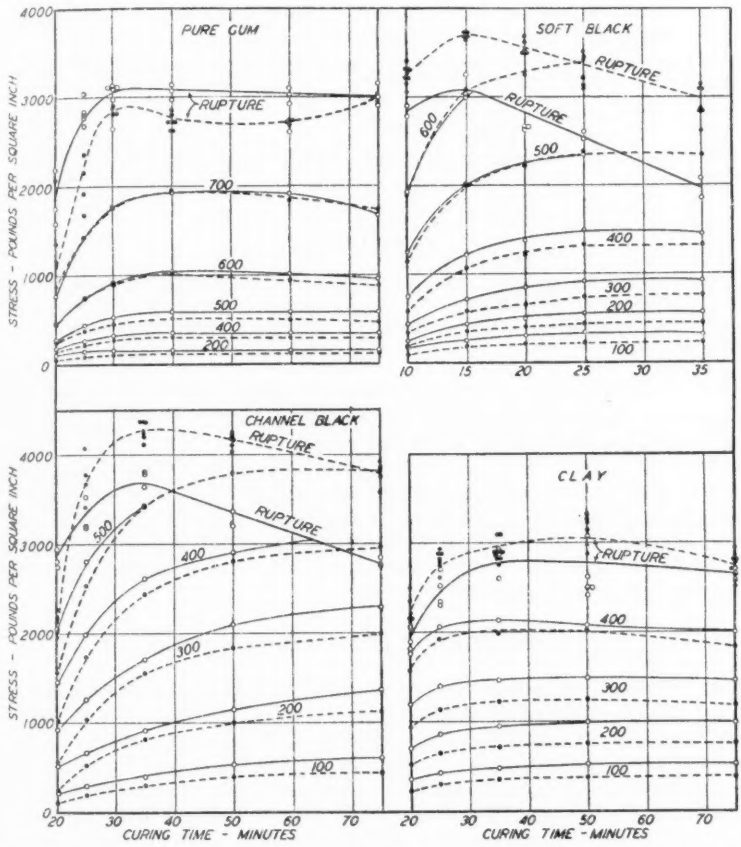

FIGURE 6.—*Stresses and tensile strengths for the two rates of stretch as a function of the time of cure.*

The solid-line and broken-line curves refer respectively to rapid and slow stretching. The numbers on the curves indicate the percent elongation. The compounds are those described in table 1.

ing to that for rapid stretching, at equal elongations, ranges from about 0.6 at 100-percent elongation to about 0.9 in the neighborhood of rupture. The relative constancy of this ratio at each elongation makes it evident that the work of extension for a given elongation obtained by rapid stretching gives no better indication of the nature of a compound than can be obtained by slow stretching.

TABLE 5.—*Comparison of work of extension of rubber compounds at optimum cure for the two rates of stretch*

Compound	Pure gum		Soft black		Channel black		Clay	
Rate of stretch	Fast	Slow	Fast	Slow	Fast	Slow	Fast	Slow
Elongation	Work of extension, inch-pounds per cubic inch							
Percent								
100	62	35	140	95	215	135	240	170
200	190	125	465	365	843	670	940	685
300	385	270	1,015	815	2,155	1,820	2,120	1,615
400	645	485	1,960	1,595	4,310	3,785	3,900	3,190
500	1,060	840	3,560	3,095	7,375	6,705	[a] 6,380	5,590
506								[a] 5,740
543					[a] 8,910	8,270		
590						[a] 10,200		
600	1,740	1,490	6,060	5,675				
605			[a] 6,210	5,835				
660				[a] 7,735				
700	3,040	2,770						
790	4,990	[a] 4,840						
820	[a] 5,540							

[a] Values at rupture.

The total work of extension to rupture for the pure-gum and clay compounds is greater for rapid stretching than for slow stretching. On the other hand, for the compounds containing soft black and channel black the work of extension is actually less for rapid stretching. Since the soft-black and channel-black compounds are known to be much more resistant to abrasive wear than the other compounds, it would seem that the work of extension to rupture is less indicative of resistance to abrasive wear when the compound is stretched rapidly than when it is stretched slowly.

V. SUMMARY AND CONCLUSIONS

Stress-strain relationships and the work of extension for four rubber compounds were studied for a rate of stretch of the order of 1,000 percent per second. This rate is sufficiently great so that the test may be considered to approach adiabatic conditions. A comparison of these data with similar data for a rate of stretch which lies in the range of speeds common to the usual routine tests shows that increased speed of stretching affects the observed tensile properties as follows: (1) The stresses are increased at elongations up to about 500 percent for the loaded compounds and up to 600 percent for the pure-gum compound, the maximum increases ranging from 75 pounds per square inch for the pure-gum compound to 245 pounds per square inch for the clay compound; (2) the stresses in the carbon-black compounds are decreased slightly at elongations near rupture; (3) the work of extension to rupture is increased for the pure-gum and clay compounds and decreased for the carbon-black compounds; and (4) the work of extension for a given elongation is increased for all the compounds. The stresses and the work of extension at the higher speed reveal no information indicative of resistance to abrasive wear that cannot be gained from similar studies of data obtained in stretching the specimen slowly.

WASHINGTON, September 13, 1939.

U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

RESEARCH PAPER RP1257

Part of Journal of Research of the National Bureau of Standards, Volume 23, November 1939

INTERNAL ABSORPTION OF GAMMA RAYS IN RADIUM-BERYLLIUM NEUTRON SOURCES

By Leon F. Curtiss

ABSTRACT

When neutron sources composed of beryllium powder mixed with a radium salt are prepared commercially, it is sometimes desirable to determine the actual amount of radium in such a source by gamma-ray comparisons with a radium standard. To do this it is necessary to know the amount of absorption of gamma rays within the source and its container.

To compute this correction, a knowledge of the absorption coefficient of the beryllium powder is required, and the equations involved are difficult to work with in the case of a cylindrical source such as is commonly used. Therefore, this internal absorption has been determined experimentally for a source contained in a brass cylinder of 8-mm internal diameter and 24.5-mm internal length with walls 1 mm thick. This cylinder contained a mixture of 1,237 mg of finely powdered beryllium and radium sulfate containing 139.7 mg of radium element. From measurements made in the usual way with a gamma-ray electroscope with 1 cm of lead filtering, the absorption correction was found to be 5.1 percent.

The determination of internal absorption of gamma radiation of radioactive substances within the radioactive salt itself can, in most practical cases, best be made experimentally. The problem has been treated mathematically by Thirring [1] and also by E. v. Schweidler [2] for the case of a sphere of radioactive material. For other configurations, such as a cylinder usually met with in practice, the computations become more difficult. An expression for the internal absorption in a cylindrical radium preparation in a glass tube of 2.66-mm internal diameter has been developed by Holuba.[3] Aside from the mathematical difficulties involved, the results of such calculations are subject to errors arising from inaccurate knowledge of absorption coefficients as well as from the fact that the simple exponential absorption law is not valid in the case under consideration.

A similar problem is presented by neutron sources consisting of a mixture of beryllium powder and a radium salt enclosed in a brass capsule. It is often desirable to determine the actual amount of radium in such a source by comparison of its gamma radiation with that from a radium standard. When comparing radium preparations of approximately equal size, the internal absorption is so nearly the same in each case that it can be neglected. This is no longer true

[1] H. Thirring, Physik. Z. **13**, 266 (1912).
[2] E. v. Schweidler, Physik. Z. **13**, 453 (1912).
[3] M. Holuba, Sitzber. Akad. Wiss. Wien [IIa] **146**, 285 (1937).

when the radium salt is mixed with a relatively large quantity of powdered beryllium.

To obtain more exact information as to the amount of internal absorption in neutron sources of this type, the measurements described below were made. Briefly stated, they consist in mixing a measured amount of radium salt with a quantity of beryllium powder, enclosing the mixture in the usual type of brass cylinder, to form a neutron source and comparing the external gamma radiation of the source thus formed with that from the same quantity of radium salt before mixing it with the beryllium.

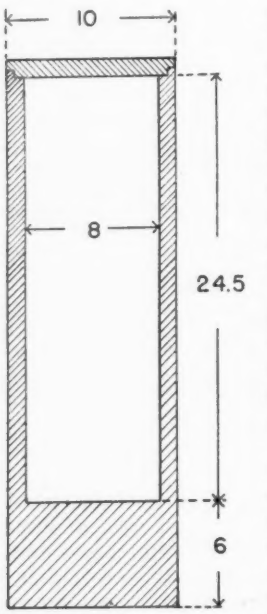

FIGURE 1.—Cross section of neutron source drawn to scale.

Dimensions are in centimeters.

A solution of radium chloride containing approximately 140 mg of radium was treated with sulfuric acid and the radium precipitated as sulfate. After carefully drying the radium sulfate it was sealed in a thin-walled glass tube and permitted to reach radioactive equilibrium. The amount of radium element was then determined by comparing its gamma radiation with that from a radium standard containing 141.5 mg of radium. The amount of radium element in the radium sulfate was thus found to be 139.7 ± 0.2 mg.

Since both the radium sulfate and the radium standard were contained in glass tubes of approximately the same wall thickness and the amounts of salt in the two were nearly the same, no correction for internal absorption is necessary.

The neutron source was prepared by mixing 1,237 mg of beryllium powder with the radium sulfate containing 139.7 mg of radium. The metallic beryllium was ground in a mortar until all of it would pass through a 60-mesh screen, after which it was thoroughly mixed with the radium sulfate. This mixture was then carefully packed into a brass cylinder, constructed as shown in figure 1, just large enough to hold the mixture. The dimensions of the cylinder are shown in the figure. The cover was soldered in position and the source permitted to reach radioactive equilibrium.

Gamma-ray measurements of this neutron source made by placing the neutron with its axis parallel to the face of an electroscope at a distance of about 150 cm, gave an apparent radium content of 132.9 ± 0.2 mg. The electroscope used for all comparisons had walls of lead 1 cm thick. The amount of filtration had some effect on the amount of absorption, but the differences were small for lead filters greater than 0.5 cm in thickness. As a result of these measurements, the absorption correction to be applied to the value for the apparent radium content of the neutron source in order to determine its actual content is 5.1 percent. This includes the correction for absorption

in the beryllium powder and in the 1-mm brass walls of the container. This correction can be expected to vary but slightly for preparations of similar shape and diameter over a range of 1 or 2 mm in either direction. Consequently, it may be used to determine the actual radium content for such sources within the usual limits of accuracy, that is, ± 0.5 percent.

WASHINGTON, October 6, 1939.

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U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

RESEARCH PAPER RP1258

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DETERMINATION OF CROSS-SECTIONAL AREAS OF STRUCTURAL MEMBERS

By James A. Miller

ABSTRACT

The cross-sectional areas of structural members are often required for computing the stresses corresponding to the applied loads. The areas of many lightweight members cannot be obtained accurately from either nominal or measured dimensions. For example, in thin-walled members small differences in thickness cause large percentage errors in area. Often the shape of a section does not permit adequate measurements of the dimensions required to calculate the area. In such cases it is often advantageous to obtain the average cross-sectional area by a volumetric method if the cross section is uniform. If the cross section varies in area from section to section the area of a given section can sometimes be obtained from the average area of a short specimen taken from that location.

The method is illustrated by a determination of cross-sectional area for a typical airship box girder, fabricated from sheet aluminum alloy. The cross-sectional area varied because of a row of flanged circular lightening holes along each side and was a minimum at sections through the centers of the holes. Specimens 0.3 inch long were taken symmetrical to a minimum section. The end surfaces were ground smooth and nominally parallel so that the lengths could be measured accurately. The volume was determined by hydrostatic weighing. A correction was applied to the average cross-sectional area of these specimens to obtain the minimum cross-sectional area. The estimated uncertainty of the determination was ±0.4 percent.

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I. INTRODUCTION

A knowledge of the cross-sectional area is frequently required in structural tests, in order to determine stresses in the structure from the external loads applied to it. In a compressive test, for example, the stress is equal to the load divided by the cross-sectional area of the specimen. It is evident, though not always appreciated, that the accuracy of the computed stress depends just as much on the accuracy of the cross-sectional area as upon the accuracy of the load.

For large structural members, cross-sectional areas computed from the nominal dimensions are usually considered adequate. For small members which have relatively compact sections of simple geometric shape the actual dimensions can be measured easily and the area can be computed readily.

Members have been built in recent years, however, which have cross sections of such size and shape that their areas cannot be obtained with sufficient accuracy from either the nominal or the measured dimensions. Members of this kind were involved when, at the request of the Bureau of Aeronautics of the Navy Department, the National Bureau of Standards undertook an investigation of the strength of aluminum-alloy airship girders of the two types shown in figure 1 and figure 2. The volumetric method for determining cross-sectional areas, described in this paper, was developed with the financial support of the Bureau of Aeronautics, Navy Department. A publication of the method seemed desirable because of its applicability to many other structural members, the areas of which cannot be obtained accurately from the usual mensuration formulas. It has been used in this Bureau to determine the cross-sectional areas of airplane wing beams, streamline tie rods, tubing, and other rolled, drawn, or extruded members.

II. METHODS OF DETERMINING CROSS-SECTIONAL AREAS

1. NOMINAL DIMENSIONS

When cross-sectional areas are computed from the nominal dimensions, the maximum difference between the cross sections of two members having the same nominal dimensions may be obtained from the specified tolerances. These tolerances frequently amount to ± 5 percent of the thickness in the case of thin sheet. Structural members of a given design formed of such sheet, as for instance the airship girder shown in figure 2, may, therefore, differ by as much as 10 percent in cross-sectional area on account of the thickness tolerances alone. Spreads in thickness as great as 6 percent of the nominal value have been found in relatively small groups of girders having the same nominal dimensions. To the effect of tolerances in thickness one must add that of tolerances in width. The maximum difference in cross-sectional area due to the width tolerances would amount to 5.2 percent in these particular girders.

FIGURE 1.—*Airship lattice girder.*

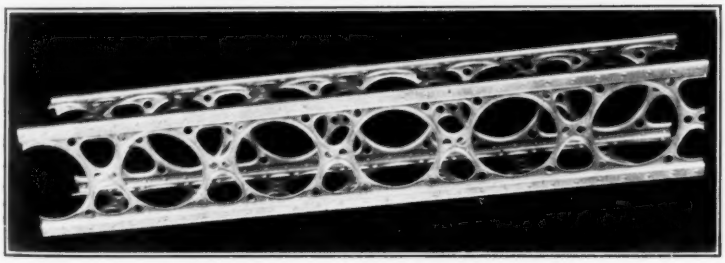

FIGURE 2.—*Airship box girder.*

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In cases where the variation from the nominal area is largely due to variation of one of the dimensions, a fairly accurate estimate of the cross-sectional area may be obtained by using an accurate measured value of this dimension and the nominal value of the other dimension.

2. MEASURED DIMENSIONS

Cross-sectional areas may be obtained directly from the measured dimensions for cross sections which are compact and of geometrically simple shape. The cross-sectional areas of members fabricated from formed sheet can sometimes be approximated by the product of the developed width of the sheet and an average value of the sheet thickness.

The following indirect method is used in this laboratory to estimate the developed widths of such sections. A strip of thin paper approximately ¼ in. wide is fitted snugly about one lateral surface of the material so that the longitudinal axis of the strip lies in the plane of the section being measured. If the surface is reentrant, a good fit may be obtained using a strip coated with a pressure-sensitive adhesive, such as transparent Scotch tape. The strip is creased at the edges between which the width is desired. Then the strip is laid out flat and the distance between the creases is measured. The width of the opposite surface may be measured in the same way. These two measurements are averaged to obtain the developed width of the section.

If the strip can be fitted around one lateral surface of the section only, the developed width may be taken as the distance between creases for this surface minus a correction $\alpha t/2$, where α is the rotation of the normal to the surface in passing from one end of the section to the other (see appendix A, fig. 7a) and t is the thickness of the sheet. For example, in measuring a closed surface on the outside, this correction amounts to πt. When the defining marks are on the outside of the strip, t should be taken as the wall thickness of the section plus the thickness of the measuring strip. However, for a thin strip the resulting error is negligible in all practical cases.

The accuracy of such a measurement of the developed width depends on the form of the edges and the practicability of fitting a strip of paper to the surface without appreciable error due to lack of contact and extension of the strip.

The accuracy of the measured values of wall thickness is an even less definite quantity. It is only rarely possible to obtain a properly weighted average thickness, taking into account variations in thickness due to forming operations. Also, observational errors, both constant and variable, in the measurements are likely to be somewhat greater for formed sheet than for smooth flat materials.

It may be concluded that the method cannot be relied upon to give accurate results for members fabricated from formed sheet metal.

3. PLANIMETER

Areas of cross sections of irregular shape may be measured by tracing a contour of the cross section with the tracing point of a planimeter. This contour may be either an impression or an enlarged photograph of the cross section.

It appears from the work of Stang, Sweetman, and Gough,[1] who applied the method to concrete-reinforcement bars, that the dimensions of the contour as computed from the area may be in error by as much as 0.01 in. For a thin section there may be appreciable errors due to burrs or bevel at the edges. The planimeter method is therefore not suitable for an accurate determination of the cross-sectional area of thin-walled sections.

4. VOLUMETRIC METHODS

(a) MEMBERS OF CONSTANT CROSS SECTION

If the cross section of a member is constant, its area can be computed by dividing the volume of the member by its length. For a long member a suitable length may be cut from the member to serve as an "area specimen."

The end surfaces of an area specimen should be plane and perpendicular to the axis of the specimen. This may require special methods of finishing the end surfaces for specimens shorter than 1 in.

The volume of an area specimen may be obtained by one of the following methods:

1. The *immersion* method.—The specimen is immersed in a liquid (preferably one having low surface tension, such as alcohol) contained in a cylindrical glass graduate. The volume of the specimen is the increase in the graduate reading due to the immersion.

2. The *weight* method.—The specimen is weighed and the mass is divided by the density.

3. The *hydrostatic-weighing* method.—The difference between the weight of the specimen and its apparent weight in a liquid of known density is determined. This difference is equal to the weight of the liquid displaced by the specimen. The mass of the liquid displaced divided by its density is therefore equal to the volume of the specimen.

The choice of the volumetric method to be used depends to some extent upon the size and shape of the specimen, the equipment available, the accuracy desired, and also the number of specimens of the same material for which the cross-sectional areas are required.

The precision of the immersion method depends to the greatest extent upon the size and shape of the specimen. If the specimen is slender and requires a relatively small volume of liquid to fill the graduate to its capacity, results can sometimes be obtained by this method which compare favorably with those obtained by the other volumetric methods.

The precision of the weight method does not depend upon the shape of the specimen or of the cross section. This is true also for the hydrostatic-weighing method, provided all adhering air bubbles are removed. Neither does the precision of these methods depend upon the cross-sectional area, provided that the specimen can be taken long enough to keep the percentage error in volume within the required limits. These methods, therefore, are especially suited to members of constant section whose area cannot be conveniently computed or measured—for example: thin-walled members, such as tubing and the chord members of airship lattice girders, and streamline rods and other rolled, drawn, or extruded members.

[1] BS J. Research **9**, 512-514 (1932) RP486.

The weight method is the most convenient method to use when the density of the material is known. Usually a specimen can be weighed with such precision that the error in volume due to the error in weight is negligible. Average values for the densities of structural materials are given in the literature; but the actual values for some alloys may differ appreciably from the average value because of differences in the composition, especially if the constituents have widely different densities. For example, for a group of 16 specimens of formed sheet aluminum alloy of a given nominal composition the difference between the highest and lowest values of density was 1.7 percent of the mean value, and for the chord members of a single girder this difference was 0.7 percent.

The hydrostatic-weighing method is generally used to obtain the volume of a solid specimen whose density is to be determined. With the apparatus and procedure to be described later, the volume of a specimen exceeding 1 cm^3 can be measured to 0.17 percent. When the density of the material is not accurately known, it is advisable to use this method either to obtain the volume of the specimen or to determine the density of the material so that the weight method can be used. The latter procedure is usually to be preferred if the volume of the specimen is too small for accurate hydrostatic weighing or if more than one volumetric determination on specimens of the same material is required.

(b) MEMBERS OF VARYING CROSS SECTION

The applicability of the weight method and of the hydrostatic-weighing method to thin-walled members of constant cross section suggests the possibility of using them to determine the minimum cross-sectional areas of thin-walled members of varying cross section—for example, airship box girders, similar to that shown in figure 2, in which the cross-sectional area of each strip of metal composing the

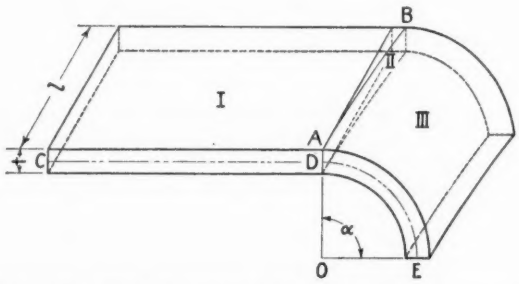

FIGURE 3.—*Hypothetical specimen for computation of excess volume.*

The following values were assumed: r, radius of AB, =1.625 in.; $t=r/32$; $OD=OE=r/8$; $\alpha=90°$; $l=0.3$ in.; $w=CD+CE=0.9$ in. The minimum cross-sectional area, $A_0=wt$. The "minimum volume" $=Al$.

sides varies because of flanged holes. Some of the flanges curve sharply from the plane of the sheet and are for the most part perpendicular to it. Other flanges are curved throughout, as indicated in figure 3. The minimum cross section between a round hole and an edge of a strip is that cut by a plane through the center of the hole and perpendicular to the longitudinal axis of the strip.

The volume of an area specimen cut from such a girder is somewhat larger than the "minimum volume" (the product of the area of the minimum cross section and the length).

The procedure for calculating the excess volume will be illustrated by a hypothetical case which imposes geometrical restrictions as severe as, and in some cases more severe than, any thus far encountered in area specimens from airship girders. This is shown diagrammatically in figure 3. The specimen may be considered as composed of three portions: I, which has constant cross section throughout; III, the flange generated by a quarter annulus, to which part of the excess volume is due; and the intermediate portion II, which is entirely an excess volume. One of the ends coincided with the minimum section, area$=A_0$, that is, it was in the plane through the center of the hole and perpendicular to the edges of the sheet. The specimen would therefore have the same relative excess as a symmetrical specimen of twice the length.

Volume III was calculated exactly by integration between the end surfaces of the specimen. A more convenient approximate solution for volume III was obtained by applying Pappus' theorem, that is, by multiplying the area of the generating quarter annulus by the length of the arc traversed by its centroid. A convenient approximate solution for the total excess volume was obtained from eq 4 and 5 derived in appendix A. All three methods gave values for total excess volume which checked within 0.01 percent of the "minimum volume." Approximate and convenient considerations such as those given in appendix A seem, therefore, adequate for computing excess volumes of area specimens of this general type.

III. PROCEDURE FOR APPLYING A VOLUMETRIC METHOD TO MEMBERS OF VARIABLE CROSS SECTION

The use of volumetric methods for determining the minimum cross-sectional area of a specimen of varying cross section requires refinements in preparing the specimen as well as consideration of the variation of cross section throughout its length. The problem involved will be illustrated by an airship box girder with circular lightening holes (fig. 2). It is hoped that the procedure followed, together with its discussion, may serve as a guide in determining the procedure for other structures.

The girder shown in figure 2 was made from four strips of aluminum-alloy sheet nominally 0.05 in. thick. The strips were flanged to form corners of the girder, two of the strips being outside at the corners and two inside. They were fastened together at the corners of the girder by rivets. Holes had been cut and flanged in each strip according to a pattern which was repeated at regular intervals throughout its length. The cross-sectional area was a minimum at sections perpendicular to the longitudinal axis of the girder through the centers of the large circular holes. For the outside strips the dimensions w, d, and f required to determine the excess volume (see appendix A) were 2.05, 3.70, and 0.09 in., respectively. For the inside strips these dimensions were 1.20, 4.55, and 0.15 in., respectively.

Let us consider the various steps in determining the minimum cross-sectional area of this girder.

1. SELECTION OF SPECIMENS

It is important that the specimen be of convenient size, large enough for accurate measurements of length and volume but not too large to obtain the minimum area accurately from the average area. A practical lower limit of length for area specimens prepared by the procedure outlined below is about 0.2 in. The upper limit of length depends upon the type of variation of section with length. It is frequently determined by the presence of irregularities, such as rivet holes, at or near the minimum section. In such cases it may be necessary to choose between a symmetrical specimen which includes the hole and an unsymmetrical specimen which avoids the hole. In general, these should be taken respectively longer and shorter than the corresponding symmetrical specimens without such irregularities. If it is necessary to include a rivet hole in a specimen, the minimum length may depend upon the size of the hole and the accuracy with which its volume may be obtained. For such a specimen it is advisable to include at least about 0.05 in. of the material on each side of the hole. It is recommended that the hole be made cylindrical with a sharp drill of known diameter before the specimen blank is cut out. The rivet hole is usually located in a relatively flat portion of the specimen, so that a few measurements of the thickness of the material in that region will give the average thickness of the material which is lacking. From these measurements the volume of the hole is computed and added to the volume of the area specimen to obtain the volume of the full specimen before dividing by the length to obtain the cross-sectional area.

For the airship girder that is used as an illustrative example, it was possible to obtain symmetrical specimens 0.3 in. long. The specimens from the outside and inside sheets had nominal volumes of 0.5 cm^3 and 0.3 cm^3, respectively. The location of the minimum cross section and identification symbols for each specimen were marked on the girder by lightly scratching with a sharp scriber.

2. DISASSEMBLING PORTION OF GIRDER CONTAINING SPECIMENS

A portion of the girder containing the specimens and extending 2 or 3 in. beyond them on each side was cut from the girder. The rivets in this portion were then removed and the parts were separated, care being taken not to mar or deform the specimens. There were then four formed strips of metal from which the area specimens were to be cut.

3. PREPARATION OF SPECIMENS

The end faces of area specimens should be perpendicular to the longitudinal axis of the member. For short specimens the end faces should, in addition, be plane and parallel in order to make possible accurate length measurements.

Each specimen was prepared by first cutting out a blank, the cuts being made normal to the longitudinal axis of the strip, and then grinding the ends until the greatest difference in length did not exceed one-half of 1 percent.

(a) CUTTING OUT BLANKS

The blanks were cut from the formed strips by two metal slitting saws mounted on the arbor of a plain milling machine. The distance between the saws was the desired length of the blank, 0.3 in., plus about 0.01 in. for finishing the ends by grinding. The strips were held in the fixture shown in figure 4. The plate, *A*, having a bar, *B*, at one end, was attached to the table of the milling machine with the bar parallel to the arbor of the machine. Clearance slots had been milled through the bar and in the plate. The blanks were cut out as follows.

The formed strip was placed in the fixture as shown in figure 4, with the minimum cross section centered between the clearance slots. The strip was clamped with one corner against the bar and the plate. The blank was then cut from the strip, the portions supported by the fixture being cut last. The other corner of the strip was then clamped in a similar position and the opposite blank was cut out.

(b) FINISHING BLANKS

The blanks were mounted in Wood's metal in a steel ring and were finished on a surface grinder equipped with a magnetic chuck.

One end of each blank was first tested for planeness on a flat plate. If the blank rocked or tilted, the end of the blank was rubbed on a wide smooth-cut mill file.

Two blanks were then arranged symmetrically about a tapped hole in the flat plate, *C*, figure 5, and clamped with the piece of sheet metal, *D*, having $\frac{1}{16}$-in. sheet rubber cemented to the lower surface. The contacts of the blanks with the plate were checked under a slight pressure before the screw was tightened.

A steel ring cut from pipe and having one end finished on a surface grinder was placed around the blanks with the finished end in contact with the plate. It was clamped by the strap, *E*.

The entire assembly was immersed in boiling water. It was removed when the temperature of the plate was higher than the melting point of the Wood's metal. Each ring was filled with Wood's metal that had been melted in a bath of boiling water. It was found desirable to have about $\frac{1}{8}$-in. clearance around the blanks so that the Wood's metal would flow freely around them.

The plate, *C*, was almost completely immersed in cold water so that the Wood's metal would solidify first at the bottom. This procedure minimized the effects of volume changes during the cooling and solidification of the molten metal.

The castings were then removed from the plate. Any projections of Wood's metal which might be touched by the grinding wheel were leveled with a heated piece of metal.

Several castings were held on the surface grinder by a magnetic chuck which had been checked to make sure that the working surface was true. A grinding wheel recommended by the manufacturer for grinding aluminum alloys was used with kerosene as the lubricant. The upper ends of the blanks were ground smooth, using feeds of about 0.001 in. and a much lighter feed for the finishing cut.

The castings were removed from the grinder and immersed in boiling water to melt the Wood's metal and release the blanks. The burrs on the ground edges were removed with a piece of aluminum

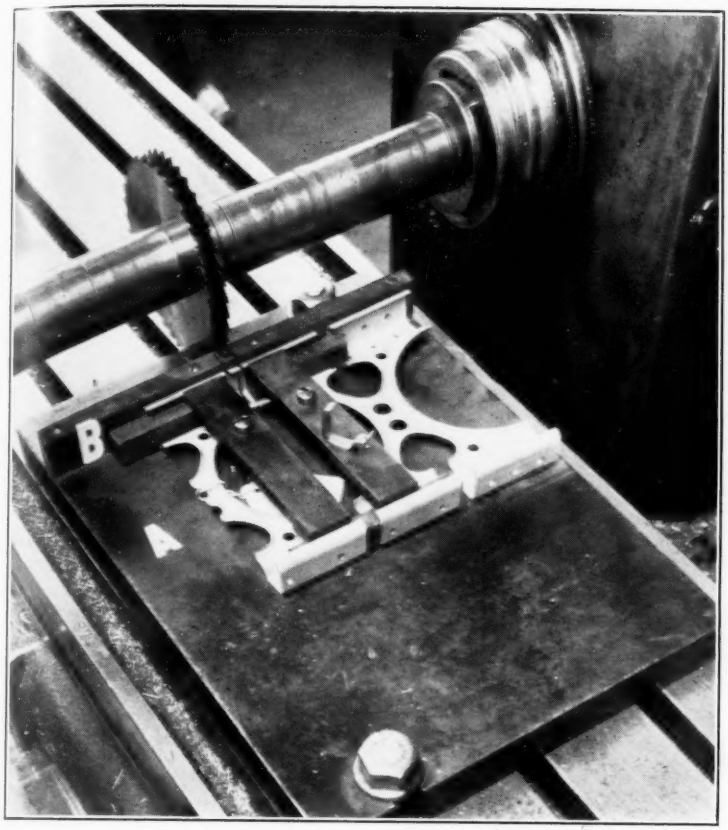

FIGURE 4.—*Cutting out blanks.*

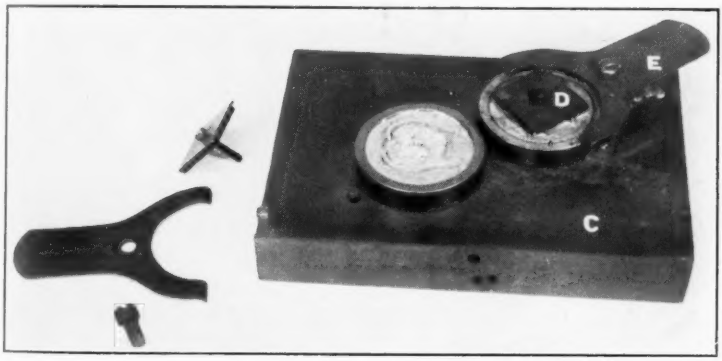

FIGURE 5.—*Blanks mounted in rings.*

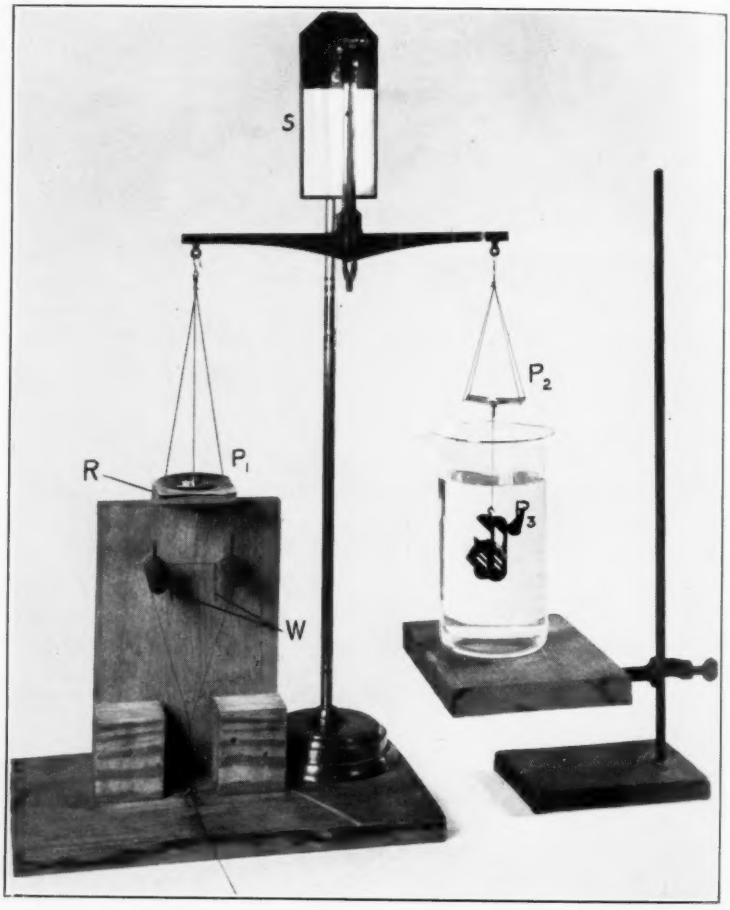

FIGURE 6.—*Mohr balance used to determine volumes of area specimens.*

alloy, care being taken not to bevel the edges, and the ground surface was lapped superficially on a roughly ground cast-iron plate. This procedure was repeated until all of the burr was removed.

The preparation of each area specimen was completed by repeating the finishing procedure for the other end of the blank.

During the whole finishing procedure it is important that the surfaces be clean when they are brought into contact, so that the blanks and castings do not become tilted.

4. DETERMINATION OF LENGTH

For measuring the length of a specimen, micrometer calipers were used. The readings were estimated to one-tenth of a division (0.0001 in.).

The length of each specimen was measured at from 10 to 15 equally spaced locations. A specimen the length of which varied more than one-half of 1 percent was reground on one end.

5. DETERMINATION OF VOLUME

The specimens were grouped together for a volumetric determination and the hydrostatic-weighing method was used. This appeared to be the most convenient procedure, since the density of the material was not accurately known and since the specimens were of the same length and the cross-sectional areas at the individual corners were not required.

In applying the hydrostatic-weighing method, the volume specimen consisting of a group of area specimens was placed on one pan of a balance and was counterbalanced. Then, while suspended from the same pan, it was immersed in a liquid of known density, and the system was balanced by adding weights to that pan.

The volume was determined with the Mohr balance shown in figure 6. Pan P_1 of the balance could be held against a pan rest, R, by two weights, W, on a cord passing through a wire loop fastened to the bottom of the pan. The cord was so arranged that when it was pulled taut, the weights were raised, allowing the beam to swing. The amplitude of the swing could be controlled by raising the weights slowly.

A pan, P_2, for holding the specimens when weighing them in air was provided for the right arm of the beam. A holder, P_3, for supporting the specimens while they were being weighed in water was suspended from the bottom of the pan P_2 by a 0.003-in. copper wire.

In order to determine the position of the pointer which passed through a slot in the support of the central bearing of the balance and consequently was least easily observed when the beam was nearly balanced, a light was so placed that the shadow of the slot and pointer could be observed on a screen, S, back of the balance.

The liquid used was distilled water. Good results were obtained by using a very fine wire for suspending the specimen holder by having the wire in the same position relative to the surface of the water for each balancing operation and by observing the following procedure.

The apparatus was assembled as shown in figure 6. The wire for suspending the specimen holder was cleaned with alcohol and examined for kinks and other irregularities. The water level was adjusted

so that that portion of the wire which passed through the surface of the water was smooth. Any air bubbles adhering to the specimen holder were removed.

The left-hand pan was held against the rest whenever specimens or weights were being changed.

The specimen was given a final cleaning with alcohol and allowed to dry, after which it was always handled with tweezers.

The temperature of the water was observed and recorded to 0.5° C.

The specimen was placed on the right-hand pan and was approximately counterbalanced by weights on the other pan. The beam was finally balanced with a 5-mg rider on the right arm of the beam. The amplitude of the swing of the beam was made very small before the final balancing by slowly raising the weights for holding the pan against the rest as the pointer neared the zero position and by manipulating the 5-mg rider. The final balancing procedure was as follows:

The 5-mg rider was placed on the right arm of the beam near the knife-edge and was then moved to the right along the beam until the right side was definitely heavy. It was then moved toward the left until the right side was definitely light. After the first trial the rider was carefully moved about one-fifth of a division at a time to keep the amplitude of swing of the beam small. The procedure was repeated until successive averages of maximum and minimum readings checked within about three-tenths of a division (0.00015 g). The average of these two values was recorded to 0.1 division.

The specimen was removed from the pan with tweezers, immersed in the distilled water, and then placed on the specimen holder after any adhering air bubbles were removed. If difficulty was experienced in removing air bubbles from the specimen, it was dipped in alcohol, then in distilled water, and then placed on the specimen holder. Weights were placed in the right pan to balance the buoyant force of the water on the specimen. An approximate balance was obtained with a 10-mg rider set at a division mark, and the final balancing was done as before with the 5-mg rider. The average reading for the 5-mg rider was recorded, also the weight represented by the 10-mg rider (0.001 g per division) and the weights in the right pan.

It was generally possible to obtain successive values for the apparent loss in weight that checked within 0.1 percent for a specimen measuring not less than 1 cm^3.

The volume was calculated from the apparent loss in weight, using factors given in the third column of table 2 in appendix B.

If the density of the specimen is desired and an analytical balance is not available, the weight in air may be obtained by balancing the system without the specimen by adding additional weights to the right pan.

6. COMPUTATION OF CROSS-SECTIONAL AREA

The average cross-sectional area of the specimen was calculated by dividing the volume by the average length. A correction computed from eq 5, appendix A, was applied to obtain the minimum cross-sectional area.

IV. DISCUSSION

1. TIME REQUIRED

The time required for determining the cross-sectional area of a girder such as the one described was found to be approximately 20 man-hours for the selection and preparation of the eight specimens treated as a group and 3½ man-hours for the determination of volume and length. The time is necessarily increased when the cross-sectional areas at the various corners are desired or when it is necessary to take specimens of different lengths.

2. ACCURACY

Since the cross-sectional area is given by the ratio of volume to length, the accuracy of its value depends upon the combined errors in the length and volumetric measurements.

The error in length will depend on the shape of the specimen and on the deviation of the end surfaces from parallelism and from perpendicularity to the longitudinal axis of the specimen. For a specimen 0.3 in. long from the airship box girder, figure 2, it was estimated from a detailed study that the average of the length measurements was between 0.16 percent less and 0.20 percent greater than the actual average length. This figure was derived as the sum of an error of from -0.03 to $+0.10$ percent due to errors in the micrometer caliper measurements resulting from deviation of the end surfaces from parallelism, an error of from -0.06 to 0 percent due to deviation from perpendicularity of the end surfaces, an error of from 0 to $+0.03$ percent due to surface irregularities, and an error of ± 0.07 percent in setting and reading the micrometer calipers.

A detailed study was also made of the possible errors in the volume determination. It was concluded that the volume obtained for a specimen or a group of specimens 0.3 in. long, having a total volume greater than 1 cm^3 (0.061 $in.^3$), was within ± 0.17 percent of the actual volume. This figure was derived as the sum of an error of ± 0.02 percent due to the tolerances for the class S[2] weights used in the determination and an error of ± 0.15 percent obtained from a statistical study of a large number of volume determinations by two observers.

The total possible error in the minimum cross-sectional area for a symmetrical specimen, without holes, of the type considered was estimated by adding the errors in length and the errors in volume and adding to these a possible error of ± 0.01 percent in the value of excess volume (see appendix A, table 1). This results in a total possible error of from -0.38 to $+0.34$ percent.

The total possible error in minimum cross-sectional area of a symmetrical specimen of the same type but with a 0.2-in. rivet hole was estimated to be from -0.70 to $+0.66$ percent due to an additional estimated error of ± 0.32 percent in determining the volume lacking because of the hole (see appendix A, table 1).

The total possible error for an unsymmetrical specimen of the same type which would avoid a 0.2-in. rivet hole having its center in the minimum section was estimated to be from -0.71 to $+0.59$ percent.

[1] See Cir. BS C3 [3d ed], 26 (1918).

The error is greater than for the symmetrical specimen of the same length because of an increase in the possible error of the excess volume (see appendix A, table 1).

V. SUMMARY

1. If a structural member has a cross section of simple geometric shape and of sufficient size for accurate measurement, the cross-sectional area can be determined readily and satisfactorily from the measured dimensions.
2. For other structural members the average cross-sectional area over a given length can be determined more accurately by volumetric methods.
3. The hydrostatic-weighing method and the weight method were found to be the most satisfactory of the volumetric methods.
4. For a structural member the cross sections of which differ in size, the minimum cross-sectional area can be determined by volumetric methods if the relationship between the minimum and the average cross-sectional areas of a suitable area specimen is known.
5. Frequently the specimen can be taken sufficiently short so that the difference between the average and minimum cross-sectional areas may be estimated or even neglected without materially affecting the accuracy of the result.
6. If short area specimens are used the end surfaces should be plane and parallel so that the length can be measured accurately.
7. The minimum cross-sectional area of a symmetrical area specimen, free from holes, 0.3 in. long, and having a volume of 1 cm^3, can be determined, by the volumetric method described, with an error of less than 0.4 percent. If there are holes near the minimum cross section, this percentage may be approximately 0.7 percent.

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VI. APPENDIX A

Derivation of Approximate Formulas for Computing the Minimum Cross-Sectional Areas of Specimens Having Flanged Circular Holes

Consider a formed strip of sheet metal such as was used in the girder, figure 2. The minimum cross section is that cut by a plane through the center of one of the large circular holes and perpendicular to the longitudinal axis of the strip.

Consider, for convenience, the material between a hole and one edge of the strip, indicated by its minimum cross section in figure 7 (a). An area specimen is taken from this region, with its end surfaces parallel to the minimum section.

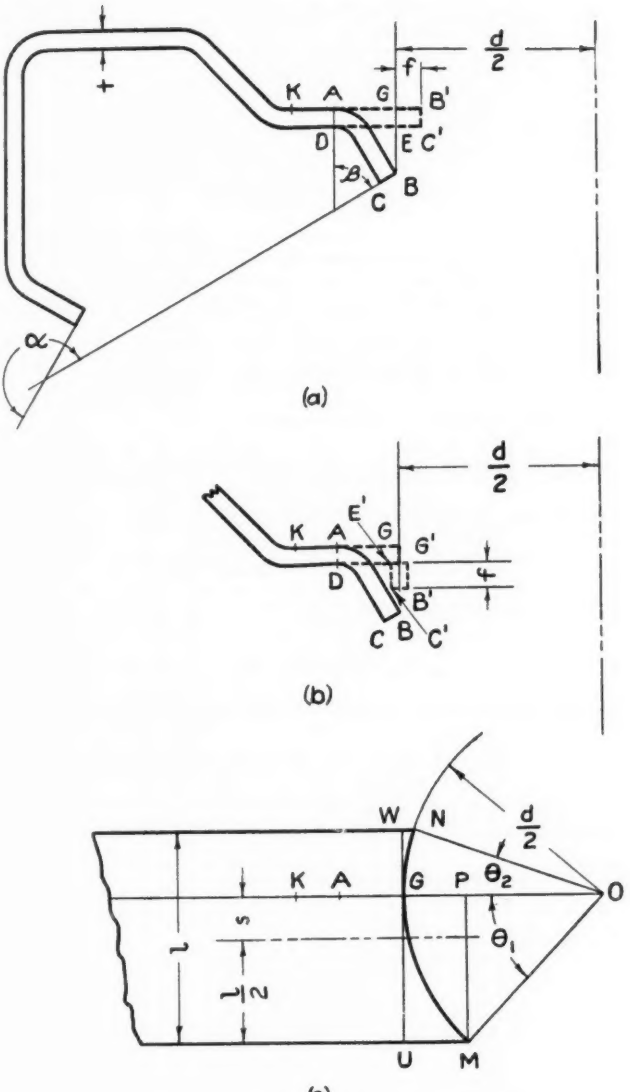

FIGURE 7.—*Assumed distribution of flange material for deriving eq 5.*

Let l=length of area specimen
d=inside diameter of hole
s=distance from minimum section to midlength of specimen

$$\theta_1=\sin^{-1}\frac{l+2s}{d} \quad (1)$$

$$\theta_2=\sin^{-1}\frac{l-2s}{d} \quad (2)$$

w=developed width of specimen. (See page 623, also fig. 7 a.)

Assume that the average thickness at and near the flange is equal to the average thickness, t, of the whole specimen.

For convenience in computation, the flange of the hole is replaced by two mutually perpendicular parts—one part (represented by its minimum cross section $AGED$ in fig. 7 a) lying in the same plane as that of the material adjacent to the flange and having an inside diameter equal to the hole diameter, d, and the other part (represented by $GB'C'E$, fig. 7 a) considered to be part of a tube (represented by the section $G'B'C'E'$, fig. 7 b) having a mean diameter equal to d and length equal to

$$f=KAB-KAG-\frac{\beta t}{2},$$

where KAB is the distance measured along the outside of the flange from a convenient point, K, near the flange, to the edge of the flange; AG is the projection of the flange upon the plane of the material adjacent to the flange; and β is the angle between the normal to AG and the edge of the flange.

The excess volume, ΔV, is equal to the total volume, V, less the "minimum volume," wtl. The excess volume involves only the material represented by the portions UGM and GWN, figure 7 (c), together with the portion of the flange replaced by a tube.

The volume represented by UGM is equal to

$$t\left[\frac{d^2}{4}(\sin\theta_1)-\frac{d^2}{8}(\sin\theta_1\cos\theta_1+\theta_1)\right]=t\left[\frac{d}{4}(l+2s)-\frac{d^2}{8}(\sin\theta_1\cos\theta_1+\theta_1)\right].$$

Similarly the volume represented by GWN equals

$$t\left[\frac{d}{4}(l-2s)-\frac{d^2}{8}(\sin\theta_2\cos\theta_2+\theta_2)\right].$$

The excess for the tubular portion equals

$$\frac{tfd}{2}(\theta_1+\theta_2)-tfl.$$

Adding these three expressions results in a total excess volume

$$\Delta V=t\left\{\frac{d}{4}\left[2l-\frac{d}{2}(\sin\theta_1\cos\theta_1+\theta_1+\sin\theta_2\cos\theta_2+\theta_2)\right]+\frac{fd}{2}(\theta_1+\theta_2)-fl\right\}.$$

If θ_1 and θ_2 are sufficiently small, as will frequently be the case, θ and $\cos\theta$ may be replaced by the first two terms of their series expansions:

$$\theta=\sin\theta+\frac{1}{6}\sin^3\theta+\frac{3}{40}\sin^5\theta+\quad\ldots$$

and

$$\cos\theta=1-\frac{1}{2}\sin^2\theta-\frac{1}{8}\sin^4\theta-\quad\ldots$$

so that

$$\Delta V=t\left\{\frac{d}{4}\left[2l-d(\sin\theta_1+\sin\theta_2)+\frac{d}{6}(\sin^3\theta_1+\sin^3\theta_2)\right]\right.$$
$$\left.+\frac{fd}{2}\left[\sin\theta_1+\sin\theta_2+\frac{1}{6}(\sin^3\theta_1+\sin^3\theta_2)\right]-fl\right\}. \tag{3}$$

Substituting in eq 3 the values for $\sin\theta_1$ and $\sin\theta_2$ given respectively in eq 1 and 2, we obtain

$$\Delta V=\frac{lt}{d}\left(1+\frac{2f}{d}\right)\left(\frac{l^2}{12}+s^2\right). \tag{4}$$

The ratio, E, of excess volume, ΔV, to "minimum volume" wtl, which is also the ratio of the excess cross-sectional area to minimum cross-sectional area, is

$$E=\frac{\Delta V}{wtl}=\frac{1}{wd}\left(1+\frac{2f}{d}\right)\left(\frac{l^2}{12}+s^2\right). \tag{5}$$

To obtain ΔE, the error in E due to an error Δs in s, substitute $s+\Delta s$ for s in eq 5 and subtract eq 5 from the resulting expression. This gives

$$\Delta E=\frac{\Delta s}{wd}\left(1+\frac{2f}{d}\right)(2s+\Delta s). \tag{6}$$

Calculations of E and of ΔE and other possible errors in the minimum cross-sectional area of the girder considered in the illustrative example are given in table 1. Calculations are also given for unsymmetrical specimens and for additional errors due to rivet holes at the minimum section.

TABLE 1.—*Calculation of excess, E, and possible errors in minimum cross-sectional area for area specimens from a box girder with circular lightening holes*

	Symmetrical specimens			Unsymmetrical specimens		
	Outside	Inside	Combined	Outside	Inside	Combined
Excess, E, eq 5:						
f, in	0.09	0.15	----------	0.09	0.15	--------
d, in	3.70	4.55	----------	3.70	4.55	--------
$1+\frac{2f}{d}$	1.049	1.066	----------	1.049	1.066	--------
$\frac{100}{d}\left(1+\frac{2f}{d}\right)=100k$	28.35	23.43	51.78	28.35	23.43	51.78
w, in	2.05	1.20	3.25	2.05	1.20	3.25
$\frac{100k}{w}$	13.83	19.52	15.93	13.83	19.52	15.93
l, in	0.30	0.30	0.30	0.30	0.30	0.30
s, in	.00	.00	.00	.25	.25	.25
$\frac{l^2}{12}+s^2$	.0075	.0075	.0075	.0700	.0700	.0700
$\frac{100k}{w}\left(\frac{l^2}{12}+s^2\right)=100E$, percent [a]	.10	.15	.12	.97	1.37	1.12
Error in locating minimum section, ΔE, eq 6:						
Δs, in	±.03	±.03	±.03	±.03	±0.03	±0.03
$\frac{100k}{w}\Delta s(\Delta s+2s)=100\Delta E$, percent [a]	+.01	+.02	+.01	+.22 −.20	+.31 −.28	+.25 −.22
Error in measurement of w:						
Δw, in	±.02	±.02	±.04	±.02	±.02	±.04
$-\frac{100E\Delta w}{w}$, percent [a]	.00	.00	.00	∓.01	∓.02	∓.01
Error due to variation in thickness:						
$-0.1\times100E$, percent [a]	−.01	−.01	−.01	−.10	−.14	−.11
Total error in estimating excess, E:						
Increase in "minimum volume," percent [a]	.01	.02	.01	.23	.33	.26
Decrease in "minimum volume," percent [a]	.01	.01	.01	.31	.44	.34
Error in computing volume of rivet holes:						
r, radius of hole, in	.1	.1	----------	--------	--------	--------
Δr, in	±.0005	±.0005	----------	--------	--------	--------
t, thickness of sheet, in	.05	.05	----------	--------	--------	--------
Δt, in	±.002	±.002	----------	--------	--------	--------
$\pi r(r\Delta t+2t\Delta r)=v$, error in volume, in.^{3}	±.000079	±.000079	±.000158	--------	--------	--------
$wtl=V$, "minimum volume," in.^{3}	.03075	.018	.04875	--------	--------	--------
$\frac{100v}{V}$, error, percent [a]	±.26	±.44	±.32	--------	--------	--------

[a] Percentage of "minimum volume."

VII. APPENDIX B

Calculation of Volume From Apparent Loss in Weight

The volume of a specimen, measured in cubic inches, can be calculated from its apparent loss in weight, measured in grams, when immersed in water, by making use of table 2. The factors in this table were calculated from standard density tables for distilled water at 15° to 35° C,[1] assuming the density of the weights to be 8.4 g/cm³ and the density of the air to be 0.0012 g/cm³. The second column of table 2 gives the volume of the specimen at the water temperature; and the third column gives the volume for duralumin specimens at 25° C, obtained from the volume at water temperature, assuming a coefficient of linear expansion of 23×10^{-6}/°C.

TABLE 2.—*Factors for conversion of apparent loss in weight in water (g) into volume (in.³)*

Water temperature	Factors for specimens at water temperature	Factors for duralumin specimens at 25° C
°C	in.³/g	in.³/g
15	0.061143	0.061185
16	.061153	.061191
17	.061163	.061197
18	.061174	.061203
19	.061186	.061211
20	.061198	.061219
21	.061211	.061228
22	.061224	.061237
23	.061239	.061247
24	.061253	.061258
25	.061269	.061269
26	.061285	.061281
27	.061302	.061293
28	.061319	.061306
29	.061337	.061320
30	.061355	.061334
31	.061374	.061349
32	.061394	.061364
33	.061414	.061380
34	.061434	.061396
35	.061455	.061413

WASHINGTON, August 26, 1939.

[1] Cir. BS C19 [6th ed.], 47 (1924).